# Naphthalene Hydrogenation over Catalysts Formed in situ from Ruthenium-Containing Thiosalts

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**Abstract**—Ruthenium-containing 1-butyl-1-methylpiperidinium thiosalts are synthesized; their decomposition in situ in a hydrocarbon medium makes it possible to form catalysts active in the hydrogenation of naph-thalene. It is shown that the modification of thiosalts with nickel leads to the formation of more active catalyst systems. A thermally stable ionic liquid 1-butyl-1-methylpiperidinium trifluoromethanesulfonate is synthesized. It is shown that the hydrogenation catalyst may be prepared by the decomposition of thiosalts in the ionic liquid and that it can be reused in several cycles without any loss in activity.

**Keywords:** ruthenium sulfide, ionic liquids, hydrogenation **DOI:** 10.1134/S0965544118140116

Among the most significant processes of modern petroleum refining are hydroprocesses, including hydrotreating which provides the removal of heteroatomic compounds from the feedstock under hydrogen atmosphere, hydrocracking, and conversion of polyaromatic compounds [1-3]. The latter plays an important role in attaining a high efficiency of feedstock conversion and is designed to improve the performance characteristics of final products [4, 5].

Traditionally, sulfides of transition metals are used an active component of catalysts for the hydrotreating of petroleum fractions. The authors of papers concerning the detailed study of activity of various sulfides in hydrogenation, hydrodesulfurization [7], and hydrodenitrogenation [8, 9] reactions showed that the highest catalytic activity is exhibited by sulfides of such metals as Ru, Rh, Os, and Ir. Owing to the fall of ruthenium prices on the world market, it became the cheapest metal of the platinum group and the most attractive for designing new hydroprocess catalysts [10]. A low ruthenium price in conjunction with its activity in the above-mentioned reactions stimulated the intense study of ruthenium sulfide as an active phase for hydrotreating catalysts. Papers published in the 1980s assisted in the ascertainment of factors affecting the properties of catalysts based on ruthenium sulfide [7, 11, 12]. The catalyst activity may be influenced by the nature of the initial reagent, synthesis technique, support type, catalyst activation conditions, and catalytic experimental conditions.

To increase the surface of the operating catalyst and the saving of the active component, metal sulfides

are frequently supported on porous compounds, such as silicates of aluminum and other metals [13, 14],  $Al_2O_3$  [13], and clays [15]. A substantially new approach to the synthesis of catalysts involves the elimination of supports during their synthesis, that is, the use of nanosized catalyst particles dispersed immediately in the hydrocarbon feedstock [16-21]. The advantage of a nanosized catalyst is the accessibility of active sites to organic molecules of any shapes and sizes. The potential disadvantage of these catalysts is their difficult separation from reaction products [17]. This problem may be solved within the framework of the approach previously developed for homogeneous metal-complex catalysts-biphasic catalysis [22, 23]. In this case, a catalyst is immobilized in a phase immiscible with products, while reaction products and initial reagents reside in another phase [24, 25]. Thus, catalyst separation and its reuse are simplified. The procedure of creating a similar catalyst system was coined the liquid phase catalyst immobilization method.

In biphasic catalysis processes, ionic liquids are applied as one of the phases used to immobilize the catalyst [26–28]. Ionic liquids are ionic organic compounds that are liquid at temperatures up to  $100^{\circ}$ C. They are stable, possess low volatility, and are easily modified through variation in the anion or cation structure [29]. Ionic liquids are flame-retardant and weakly toxic; they have a wide temperature range of liquid aggregate state ( $300-400^{\circ}$ C) [30]. In recent years, ionic liquids have been advantageously applied



Fig. 1. Schematic of synthesis of precursors Ni/[BMPip] $Ru_x S_y$ : (a) P2, (b) P3, (c) P1–Ni, and (d) [BMPip] $Ru_x S_y$  (P1).

for the synthesis of nanosized inorganic compounds [31-34].

It should be emphasized that variation in the cation and anion structure enables the synthesis of ionic liquids in which polyaromatic hydrocarbons are highly soluble and in which nanoparticles of the active component, for example, sulfide, can be immobilized [35]. The product resulting from the hydrogenation process-a naphthene-aromatic hydrocarbon-on the contrary, is poorly soluble in an ionic liquid and during the reaction forms a phase immiscible with it. As a result, a high selectivity for these products is attained and the catalyst system ionic liquid-active component may be easily reused many times. No data are available on the use of alternative solvents in systems with hydrogenation catalysts based on nanosized ruthenium sulfide. The goal of this work was to study the process of hydrogenation over ruthenium-containing catalysts formed in situ in the reaction medium from precursors, namely, thiosalts, including their immobilization in the ionic liquid.

#### EXPERIMENTAL

# Synthesis of Catalyst Precursors and Ionic Liquid

**1-Butyl-3-methylpiperidinium bromide** was synthesized as described in [36].

**Precursors** were synthesized according to schemes outlined in Fig. 1.

Synthesis of precursor [BMPip] $Ru_xS_y$  (P1). To a solution containing 0.002 mol of  $RuCl_3 \cdot xH_2O$  and 20 mL of acetone, a solution of 1-butyl-1-methylpiperidinium bromide (BMPipBr) containing 0.006 mol BMPipBr and 20 mL of acetone/water mixture (1 : 1, vol/vol) was added dropwise, and the resulting mixture was stirred for 30 min.

Synthesis of precursor Ni/[BMPip]Ru<sub>x</sub>S<sub>y</sub> (P2). To a solution containing 0.002 mol of RuCl<sub>3</sub> · xH<sub>2</sub>O and 20 mL of acetone, a solution containing 0.00123 mol of NiCl<sub>2</sub> · 6H<sub>2</sub>O and 10 mL of acetone/water mixture (1 : 1, vol/vol) was added dropwise. To the as-obtained solution, a solution containing 0.006 mol of BMPipBr and 20 mL of acetone/water mixture (1 : 1, vol/vol) was added dropwise, and the resulting mixture was stirred for 30 min.

Synthesis of precursor Ni/[BMPip]Ru<sub>x</sub>S<sub>y</sub> (P3). To a solution containing 0.00123 mol of NiCl<sub>2</sub> ·  $6H_2O$  and 10 mL of acetone/water mixture (1 : 1, vol/vol), a solution containing 0.006 mol of BMPipBr and 20 mL of acetone/water mixture (1 : 1, vol/vol) was added. The as-obtained solution was added dropwise to the solution containing 0.002 mol of RuCl<sub>3</sub> ·  $xH_2O$  and 20 mL of acetone, and the resulting mixture was stirred for 30 min.

Synthesis of precursor Ni/[BMPip]Ru<sub>x</sub>S<sub>y</sub> (P1–Ni). To a solution containing 0.002 mol of RuCl<sub>3</sub> · xH<sub>2</sub>O and 20 mL of acetone, a solution containing 0.006 mol



Fig. 2. Schematic of synthesis of 1-butyl-1-methylpiperidinium trifluoromethanesulfonate.

of BMPipBr and 20 mL of acetone/water mixture (1:1, vol/vol) was added dropwise, and the resulting mixture was stirred for 30 min. Ten milliliters of acetone was added, the mixture was stirred for another 30 min, and  $H_2S$  was blown for 2 h at room temperature under stirring. To the as-obtained resulting crystalline substance, 20 mL of solution containing 0.00123 mol of NiCl<sub>2</sub> · 6H<sub>2</sub>O and 10 mL of acetone/water mixture (1:1, vol/vol) were added, and the resulting mixture was stirred for 30 min.

In the synthesis of all precursors after completion of all the above-described procedures, 10 mL of acetone was added, the resulting mixture was stirred for 30 min, and  $H_2S$  was blown for 2 h at room temperature under stirring. The obtained precipitates were rinsed with isopropyl alcohol (3 × 10 mL) and diethyl ether (1 × 5 mL) and dried in air.

Synthesis of 1-butyl-3-methylpiperidinium trifluoromethanesulfonate ([BMPip]CF<sub>3</sub>SO<sub>3</sub>) (Fig. 2). [BMPip]Br (0.1 mol) was dissolved in 50 mL of distilled water, potassium trifluoromethanesulfonate  $KSF_3SO_3$  (0.1 mol) was added, and the resulting mixture was stirred at a temperature of 80°C for 1 h. The reaction mixture was cooled to room temperature, 50 mL of dichloromethane was added, and the obtained mixture was stirred at room temperature for 8 h. The final mixture was separated into phases, and the ionic liquid was extracted from the aqueous phase with dichloromethane (4  $\times$  10 mL). The combined organic phase was rinsed with distilled water (3  $\times$ 10 mL) and dried over magnesium sulfate. After 12 h, MgSO<sub>4</sub> was filtered off, and the solvent was distilled off at a temperature of 40°C. The produced sample was vacuum dried a temperature of 90°C. Yield, 93%.

The purity and structure of the products were studied by <sup>1</sup>H NMR spectroscopy. <sup>1</sup>H NMR (**CDCl**<sub>3</sub>):  $\delta 0.89$  (t, 3H, CH<sub>2</sub>CH<sub>3</sub>), 1.3 (m, 2H, CH<sub>2</sub>CH<sub>3</sub>), 1.8

# (m, 8H, **CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>)**, 2.9 (s, 3H, **NCH<sub>3</sub>**), 3.3 (m, 6H, N(**CH<sub>2</sub>**)<sub>3</sub>) ppm.

#### Physicochemical Analysis of Precursors, Catalyst, and Ionic Liquid

The contents of carbon, hydrogen, nitrogen, and sulfur in organic compounds were determined by the Pregl–Dumas technique on a CHNS/O EA1108 Elemental Analyzer instrument (Carbo Erba). The ruthenium and nickel contents were measured by atomic adsorption spectroscopy on an AAnalyst 400 instrument (PerkinElmer).

The elemental analysis of the synthesized precursors (Table 1) shows that for precursor P1 the S/Ru atomic ratio is 2.63 and for precursors P3 and P1–Ni the S/Ru atomic ratios are almost equal (2.7–2.9); the sulfur content in sample P2 is lower (the S/Ru atomic ratio is ~2.1). According to the techniques of precursor synthesis, the content of reaction mixture components was chosen so that the Ni/Ru atomic ratio in final materials was 0.6; however, this value is inconsistent with the elemental analysis of the samples (Table 1). The highest amount of nickel is observed for sample P1–Ni and its Ni/Ru atomic ratio is 0.15.

Sample [BMPip]CF<sub>3</sub>SO<sub>3</sub> was studied on an IFS 66 v/S IR spectrometer (Bruker Optics) (Fig. 3). IR spectra were registered in the transmission mode at room temperature and every 20°C in the range of 60–200°C (50 scans; resolution, 2 cm<sup>-1</sup>; range, 400–4000 cm<sup>-1</sup>). The mathematical processing of the spectra was performed using the OPUS-7 software package. The most intense bands in the spectra correspond to vibrations of C–F, S–O, and S=O bonds in trifluoromethanesulfonium anions. Less intense bands at 1000–1300 and 550–850 cm<sup>-1</sup> are due to mixed skeletal vibrations in alkyl-substituted piperidine ligands; in

Precursor	Designation	С	S	Н	Ν	Ru	Ni
$[BMPip]Ru_xS_y$	P1	13.4	38.6	2.3	1.6	44.1	_
Ni/[BMPip]Ru <sub>x</sub> S <sub>y</sub>	P2	11.6	33.40	2.50	0.5	51.3	0.7
	P3	12.9	39.80	2.40	0.7	43.2	1.0
	P1–Ni	12.6	38.90	2.8	0.3	41.8	3.6

Table 1. Elemental analysis of precursors



Fig. 3. Survey IR spectrum of [BMPip]CF<sub>3</sub>SO<sub>3</sub> and fragmented spectra recorded in the temperature range of 25–200°C.

addition, bands due to the stretching and bending vibrations of C–H bonds are well defined in the spectra. The presence of bands at 1640 and  $3450 \text{ cm}^{-1}$  indicates that the sample contains water which may reside in hygroscopic KBr used in sample preparation for analysis rather than in the ionic liquid itself. A more detailed analysis of the spectra measured in the temperature range of 25–200°C reveals that during heating the intensity of the bands decreases. This is evidence for small structural rearrangements occurring in the structure of ionic liquid under heating. Upon cooling all bands reassume their shapes. Water in this system almost fully desorbs upon heating to 100°C and reappears the sample upon cooling in air.

The thermal stability of [BMPip]CF<sub>3</sub>SO<sub>3</sub> was analyzed on a SDT Q600 derivatograph (TA Instruments). Weight loss curves and heat effects were registered under linear temperature rise at a rate of  $10^{\circ}$ C/min in the range of  $20-1000^{\circ}$ C. The data were recorded and processed using the Universal Analysis software program. According to the thermal analysis, the onset temperature of [BMPip]CF<sub>3</sub>SO<sub>3</sub> decomposition was 362°C. This fact indicates that this compound can be used without degradation in the high-temperature hydrogenation of aromatic hydrocarbons.

The <sup>1</sup>H NMR spectra of  $[BMPip]CF_3SO_3$  were measured at room temperature on a MSL 300 instru-

ment (Bruker) operating at a frequency of 300 MHz; the magnetic intensity was 7 T.

The structure and morphology of the catalysts isolated after the reaction were studied by high-resolution transmission electron microscopy (TEM) on a JEM 2100 microscope (JEOL) at an accelerating voltage of 200 kV.

## Catalytic Tests and Products Analysis

Catalytic tests on hydrogenation were run at an increased pressure and intense stirring of the reaction mixture in a steel autoclave under hydrogen atmosphere. The catalyst system was prepared according to the following order of operations. The autoclave was loaded with the catalyst precursor and 10% naphthalene solution in benzene. When the precursor in the ionic liquid was applied, it was dissolved using a LUZD-1.5 lab ultrasonic disperser. The ionic liquid/substrate ratio was 1 : 1 (wt/wt). The autoclave was pressurized, filled with hydrogen to a pressure of 5.0 MPa, and kept for 5 h at 350°C.

Hydrogenation products were analyzed on a Kristallyuks-4000M chromatograph equipped with a flame-ionization detector and a SPB-1 capillary column (Supelco) with a stationary liquid phase (dimensions,  $30 \text{ m} \times 0.25 \text{ mm}$ ; carrier gas, helium). Conver-

Sulfiding	$n_{(\text{naphth})}/n_{(\text{Ru})}$		Selectivity, %	Naphthalene	SCA h <sup>-1</sup>	
		trans-decalin	cis-decalin	tetralin	conversion, %	SCA, II
_	105.3	_	_	100	0.2	0.01
2.5 wt % S	105.3	3.2	1.2	95.6	12.9	5.8
	56.3	1.4	0.4	98.2	40.3	9.3
	7.3	13.2	8.4	78.4	94.4	3.6

**Table 2.** Naphthalene hydrogenation using precursor  $[BMPip]Ru_xS_v$  (P1)

sion was calculated as the degree of conversion of initial naphthalene to decalins and tetralin.

The activities of catalyst systems were compared in terms of a conventional parameter—specific catalytic activity (SCA,  $h^{-1}$ ), which was calculated according to the formula

$$SCA = \frac{(5KS_{\rm D} + 2KS_{\rm T})}{tn_{\rm (Ru)}}$$

where *K* is the naphthalene conversion; *S* is the selectivity for decalin (D) and tetralin (T);  $n_{\text{(naphth)}}$  is the naphthalene content in the initial feedstock, mol;  $n_{\text{(Ru)}}$  is the ruthenium content, mol; and *t* is the reaction time, h.

#### **RESULTS AND DISCUSSION**

Table 2 lists on the data on naphthalene hydrogenation over catalysts obtained by in situ decomposition of precursor  $[BMPip]Ru_xS_y$  (P1) containing no Ni. As is seen, naphthalene hydrogenation proceeds only after introduction of additional elemental sulfur. Probably, this procedure provides formation of pyritelike structure  $RuS_2$  under the used reaction conditions; in accordance with [37], this structure is active in the hydrogenation reaction.

Naphthalene conversion linearly depends on precursor concentration in the feedstock (Table 2). At  $n_{(naphth)}/n_{(Ru)} = 105.3$  (mol/mol), tetralin is the main reaction products; the amount of decalins in the system is less than 5%. As the molar ratio of naphthalene/ruthenium is decreased to 7.3, the naphthalene conversion grows and attains 94.4%. Tetralin remains the main product, but now the amount of decalins is above 20%; however, the value of specific catalytic activity decreases.

In Fig. 4, the activities of the catalysts prepared via the decomposition of Ni/[BMPip]Ru<sub>x</sub>S<sub>y</sub> precursors (P2, P3, P1–Ni) containing Ni are compared. Comparison was conducted at a molar ratio of  $n_{(naphth)}/n_{(Ru)} = 105.3$ . As is seen, in the absence of sulfur additives, almost no naphthalene hydrogenation proceeds for all the used precursors. A marked conversion of naphthalene is observed only in the case of precursor P3, but it is as low 7%. Upon addition of ele-

mental sulfur, hydrogenation proceeds in the case of all precursors.

For all the tested precursors, upon a rise in their concentration in the feedstock, the conversion of naphthalene grows. At a molar ratio of naphthalene/Ru = 7.3, the degree of conversion is almost the same (99%) for all of the precursors. As the naphthalene/Ru ratio increases, the conversion of naphthalene/Ru = 105.3, the highest naphthalene conversion is achieved in the case of precursor P1–Ni (37.6%). For all precursors, tetralin is the main product of hydrogenation (Table 3).

Thus, to obtain ruthenium sulfide active in the hydrogenation of aromatic compounds by the situ decomposition of precursors  $[BMPip]Ru_xS_y$  and  $Ni/[BMPip]Ru_rS_v$ , the introduction of additional elemental sulfur is required. The lowest activity is exhibited by the catalyst synthesized by the in situ decomposition of precursor [BMPip] $Ru_x S_v$  (P1), in which Ni is absent. The incorporation of Ni into the composition of the precursors improves their catalytic activity, in good agreement with the published data [38]. The highest activity is shown by precursor P1-Ni which is by Ni deposition prepared on precursor  $[BMPip]Ru_{x}S_{y}$  already containing sulfur (Table 3). According to elemental analysis, just for this precursor, the Ni/Ru ratio is the highest, namely, 0.15. It

![](_page_4_Figure_15.jpeg)

**Fig. 4.** Effect of sulfur additives on the activity of catalysts synthesized by in situ decomposition of precursors.

Precursor	$n_{(\text{naphth})}/n_{(\text{Ru})}$		Selectivity, %	Naphthalene	SCA h <sup>-1</sup>	
		trans-decalin	cis-decalin	tetralin	conversion, %	SCA, II
P2	105.3	0.8	0.2	99.0	18.8	8.0
	56.3	0.3	0.4	99.3	48.3	11.0
	7.3	8.5	12.8	78.7	99.5	3.8
P3	105.3	0.4	4.0	95.6	10.0	4.5
	56.3	1.5	1.0	97.5	51.8	12.1
	7.3	4.0	7.6	88.4	99.3	3.4
P1-Ni	105.3	0.7	0.4	98.9	37.6	16.1
	56.3	0.5	0.8	97.8	78.8	17.9
	7.3	4.5	3.2	92.3	99.0	3.2

**Table 3.** Naphthalene hydrogenation using precursors Ni/[BMPip]Ru<sub>x</sub>S<sub>y</sub> (P2, P3, and P1–Ni)

appears that an increase in the nickel content in the samples affords production of a catalyst possessing a higher activity in naphthalene hydrogenation to tetralin.

The TEM analysis of the P1–Ni  $(n_{(naphth)}/n_{(Ru)} =$  7.3) catalyst isolated after the reaction (Fig. 5) suggests that particle aggregates having sizes of 30–200 nm are formed; at a larger magnification, nanostructures are observed. For particles with sizes of 15–18 nm, an interplanar distance of 0.61 nm corresponds to plane (011) of phase Ni<sub>17</sub>S<sub>18</sub> [39]; in addition, the micrographs indicate the presence of finer (6–11 nm) particles, for which an interplanar distance of 0.28 nm, in accordance with [40], corresponds to plane (200) of the RuS<sub>2</sub> phase.

The catalyst system based on sulfide particles dispersed in the ionic liquid was synthesized using the most active Ni/[BMPip]Ru<sub>x</sub>S<sub>y</sub> (P1–Ni) precursor. Even though the highest SCA was observed at a molar

ratio of  $n_{\text{(naphth)}}/n_{\text{(Ru)}} = 56.3$ , tests were conducted using  $n_{\text{(naphth)}}/n_{\text{(Ru)}} = 7.3$  because of a higher content of decalin in conversion products. A noticeable reduction in naphthalene conversion in the presence of ionic liquid [BMPip]CF<sub>3</sub>SO<sub>3</sub> (Fig. 6) may be explained by the partial blocking of the catalyst surface

by  $CF_3SO_3^-$  anions and the formation of  $\pi$ -cationic complexes  $[BMPip]^+$ -naphthalene. The latter phenomenon may decrease the electron density on the substrate and causes reduction in the rate of hydrogenation.

Additionally, a series of tests were conducted to access the possibility of the repeated use of the catalyst system. The catalyst system was separated from reaction products through simple separation of immiscible phases. It was found that the time-on-stream stability of the system is preserved in four cycles as a minimum. It should be noted that selectivity parameters for conversion products also remain unchanged (Fig. 6).

![](_page_5_Figure_9.jpeg)

Fig. 5. TEM micrographs of the precursor P1-Ni-based catalyst isolated after the reaction.

![](_page_6_Figure_1.jpeg)

Fig. 6. Comparison of the activities of catalysts formed from precursor P1–Ni and precursor in ionic liquid.

Thus, we showed that the degree of conversion of naphthalene and the selectivity of hydrogenation for the catalysts formed in situ from  $[BMPip]Ru_xS_y$  and Ni/ $[BMPip]Ru_xS_y$  precursors depend on the method of preparing precursors. Sulfide ruthenium catalysts were synthesized in situ in a thermally stable ionic liquid, and it was shown that they can be reused in the reaction of naphthalene hydrogenation without any loss in activity.

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