

Ship-in-bottle synthesis of Pt–Rh carbonyls in NaX and NaY: FTIR study

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Received 3 July 2000; received in revised form 11 October 2000; accepted 11 October 2000

Abstract

$[\text{Pt}(\text{NH}_3)_4]^{2+}$ and $[\text{Rh}(\text{NH}_3)_5\text{Cl}]^{2+}$ were ion exchanged into NaX or NaY either individually or simultaneously in Pt/Rh molar ratios 1:1 and 1:5. Carbonylation of the Pt ammine in NaX at 75–100°C leads to the orange $[\text{Pt}_3(\text{CO})_6]_2^{2-}$ carbonyl and that of Rh ammine to the greenish $[\text{Rh}_6(\text{CO})_{15}]^{2-}$. Under the same reaction conditions, the mixture of both amines (1:5) reacts to an almost white or pale beige bimetallic carbonyl, assigned to $[\text{PtRh}_5(\text{CO})_{15}]^-$. This is also formed from the (1:1) mixture with the excess of Pt ammine carbonylated to $[\text{Pt}_3(\text{CO})_6]_2^{2-}$. The same bimetallic monoanionic carbonyl is formed in less basic NaY, although Rh ammine alone reacts in this matrix very slowly only to the neutral $\text{Rh}_6(\text{CO})_{16}$ complex. Oxidation of $[\text{PtRh}_5(\text{CO})_{15}]^-$ at 100°C extracts some $\text{Rh}(\text{CO})_2^+$ from the bimetallic carbonyl, but fast recarbonylation to the original species occurs. © 2001 L. Brabec. Published by Elsevier Science B.V. All rights reserved.

Keywords: Pt–Rh carbonyls; FTIR; Zeolites

1. Introduction

Supercages of NaX and NaY have been often used as microporous carriers of anionic Pt carbonyls [1–5]. The hexanuclear $[\text{Pt}_3(\text{CO})_3(\mu_2\text{-CO})_3]_2^{2-}$ carbonyl is formed easily and selectively in NaX [4,5]. Syntheses of polynuclear Pt anionic carbonyls in NaY [1–3] and in other types of zeolites (EMT [6], FSM [7,8]) have been published. In opposite, the synthesis of anionic Rh carbonyls in zeolites have not been well documented to date. Supported neutral $\text{Rh}_6(\text{CO})_{16}$ complex has been reported on NaX [9] and mainly on NaY [10–14].

Neutral $\text{Rh}_4(\text{CO})_{12}$ was also detected in NaY [12,15]. Some undefined Rh anionic complex was assumed to be formed in NaY the basicity of which was increased by treating with NaN_3 [16]. We have prepared a greenish Rh carbonyl in NaX and assigned it to $[\text{Rh}_6(\text{CO})_{15}]^{2-}$ [17]. This study has been aimed

to synthesise bimetallic Pt–Rh carbonyls by carbonylation of mixtures of Pt and Rh amines in NaX and NaY. Literature data concerning the IR spectra of bimetallic Pt–Rh carbonyls prepared in solutions and anchored on oxides are listed in Table 1. An excess of Rh seems to be necessary for their stability as follows from the composition of higher anionic Pt–Rh carbonyls: PtRh_6 , PtRh_8 , PtRh_{12} , Pt_2Rh_9 , $\text{Pt}_2\text{Rh}_{11}$, $\text{Pt}_4\text{Rh}_{18}$ with 16–35 CO ligands and two to four negative charges [18]. $[\text{PtRh}_5(\text{CO})_{15}]^-$ was successfully synthesised on MgO (Table 1); no data are available, to our knowledge, on the preparation of bimetallic Pt–Rh carbonyls in zeolites.

2. Experimental

2.1. Zeolites

NaX (Si/Al = 1.25) was supplied by Serva International, Heidelberg and NaY (Si/Al = 2.5) by

Table 1
IR wave-numbers of C–O bonds in anionic Pt–Rh carbonyls^a

Complex	Wave-number (cm ⁻¹)	Colour	Reference
[PtRh ₂ (CO) _x] ₂ ⁻	THF: 2000s ^b , 1956s , 1810m, 1790m, 1735w	Yellow-brown	[20]
[PtRh ₄ (CO) ₁₂] ₂ ⁻	THF, PPN salt: 2015w, 1982s , 1947s , 1812s , 1788m		[20]
[PtRh ₄ (CO) ₁₄] ₂ ⁻	THF, PPN salt: 2030w, 1995s , 1962s , 1807m, 1750m MeOH, Na ⁺ : 2006s , 1973s , 1813m, 1775m	Yellow-orange	[20] [20]
[PtRh ₅ (CO) ₁₅] ⁻	THF, [(Ph ₃ P) ₂] ⁺ : 2082w, 2038s , 2011m, 1791ms THF, PPN salt: 2078w, 2034s , 2004m, 1785ms; MeOH (THF)/MgO: 2082vw, 2040s , 2009m, 1789m PPN salt: 2076m, 2041s , 2008sh, 1791m MeOH: 2082vw, 2041s , 2009m, 1790ms MgO: 2082vw, 2041s , 2015ms, 1812br hydr. NaX: 2051s , 2028m(sh), 1758m mildly dehydr. NaX: 2048s , 2028sh, 1762m hydr. NaY: 2065s , 2035m(sh), 1752ms	Brown Brown Dark brown Almost white Pale beige Pale beige	[19] [20] [21] [21] [22] [22] This work

^a Intensities: s: strong; m: medium; w: weak; sh: shoulder; br: broad; vw: very weak.

^b The numbers in bold hold for the main characteristic bands.

VURUP, Slovak Republic. The sodium cations were partially ion exchanged for [Pt(NH₃)₄]²⁺ and/or [Rh(NH₃)₅Cl]²⁺ from dichlorides (Aldrich) at room temperature for 48 h to get 6.6 wt.% Pt, 3.5 wt.% of Rh and the same molar amount of (Pt + Rh) in ratios 1:1 and 1:5. The parent amines in zeolites are denoted as PtX, RhX, PtY, RhY, PtRhX and PtRhY.

2.2. Carbonylation procedure

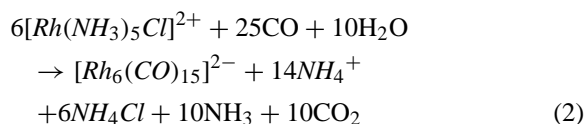
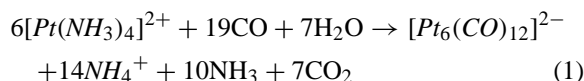
The samples were evacuated in situ before measurements usually at 25°C for 1 min (only to remove the air, samples denoted as hydrated). Mild dehydration is related to evacuation at 60°C for 5 min. A value of 500 mbar of CO (Linde, 99.97%) were allowed to react with PtX, RhX and PtRhX at 75 or 100°C, with PtY, RhY and PtRhY at 100°C, for at least 18 h.

IR spectra were measured in situ during carbonylation using a Nicolet Protégé 460 FTIR spectrometer with a DTGS detector (resolution 4 cm⁻¹). Weight of the zeolite wafers was 7–9 mg cm⁻². These wafers were heated directly in the section of IR beam, the background spectra were measured by lifting the sample to the upper part of the infrared cell.

3. Results

PtX and RhX samples exhibit an almost complete carbonylation after 15 h, if the temperature was held at

100°C (Fig. 1) and after 48 h, if the sample is heated at 75°C (not shown; the final spectra are virtually identical with those in Fig. 1). The extent of carbonylation can be seen in insets where the loss of bands belonging to N–H deformation vibrations in NH₃ ligands is shown (1372–1352 cm⁻¹ for Pt and 1340 cm⁻¹ for Rh). The band around 1460 cm⁻¹, increasing in all cases, corresponds to NH₄⁺ cations compensating the zeolite charge (Eqs. (1) and (2)); formulas in italics hold for zeolite-encaged compounds).



Carbonylation of PtX leads to the orange dianionic complex [Pt₃(CO)₃(μ₂-CO)₃]₂²⁻ with characteristic bands at 2048 and 1778 cm⁻¹. The former band (terminal CO) is accompanied by a shoulder 2081 cm⁻¹, the latter band (bridged CO) by a shoulder 1804 cm⁻¹ reflecting a mild deficit of water (the complete shift of bands at 1778 → 1806 and 2048 → 2024 cm⁻¹ appears only after strong dehydration before carbonylation [5]).

Carbonylation of RhX gives a greenish complex with characteristic bands at 2022 (with a shoulder at

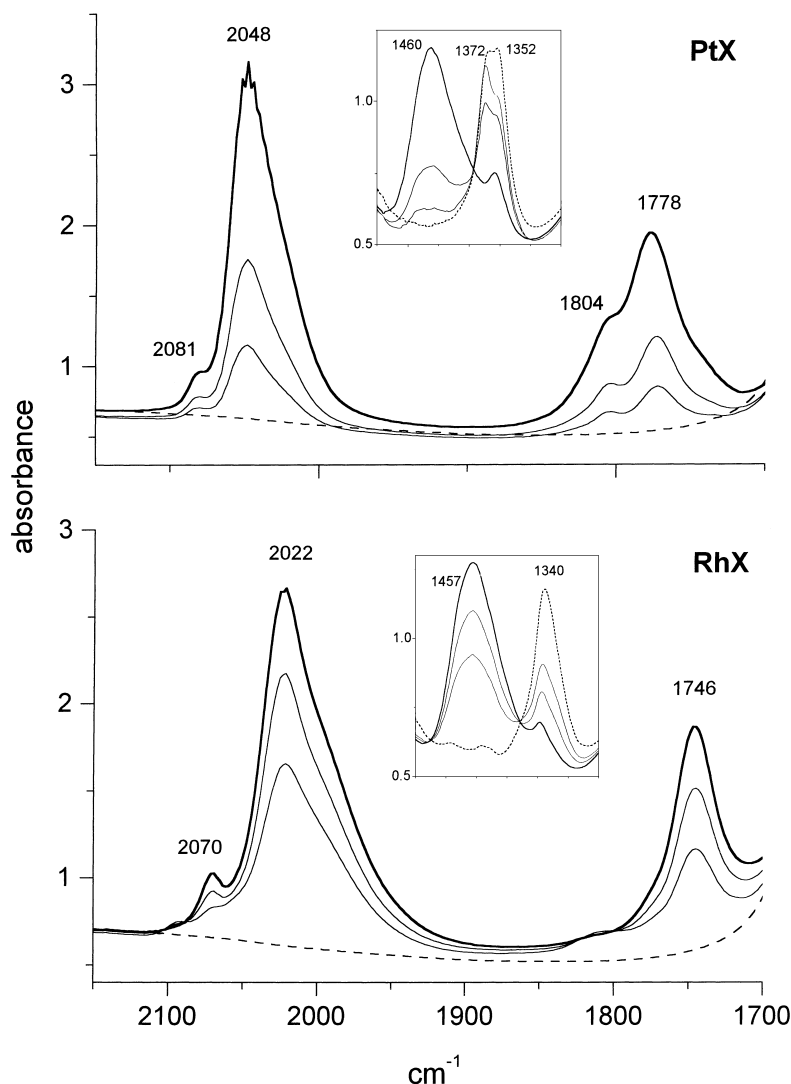


Fig. 1. Carbonylation of PtX and RhX at 100°C. Top-PtX and bottom-RhX spectra after: dashed 2 min, solid 2 and 4 h, bold solid 15 h. The same holds for insets.

2070 cm^{-1}) and at 1746 cm^{-1} . These bands were assigned to the dianionic complex $[\text{Rh}_6(\text{CO})_{15}]^{2-}$. The carbonylation rate is a little higher than that with PtX.

If these samples were mildly dehydrated and carbonylated at 75°C, the formation of polynuclear complexes becomes slower, CO bands develop for a longer time (ca 3 days). For Pt ammine, the band at 1352 shifts to 1380 cm^{-1} due to the distortion of the ammine symmetry (not shown, reported in [1,5]). The

NH_3 band in RhX shifts to 1315 cm^{-1} only after strong dehydration, however, a small water deficit is sufficient to suppress the formation of polynuclear Rh carbonyls. After 12 h, only $\text{Rh}(\text{CO})_2^+$ carbonyl is present (characteristic bands at 2095 and 2015 cm^{-1}) together with unreacted primary Rh ammine complex (ca 1/2 of the original amount). A slow agglomeration of this Rh dicarbonyl to $[\text{Rh}_6(\text{CO})_{15}]^{2-}$ proceeds for about 3 days.

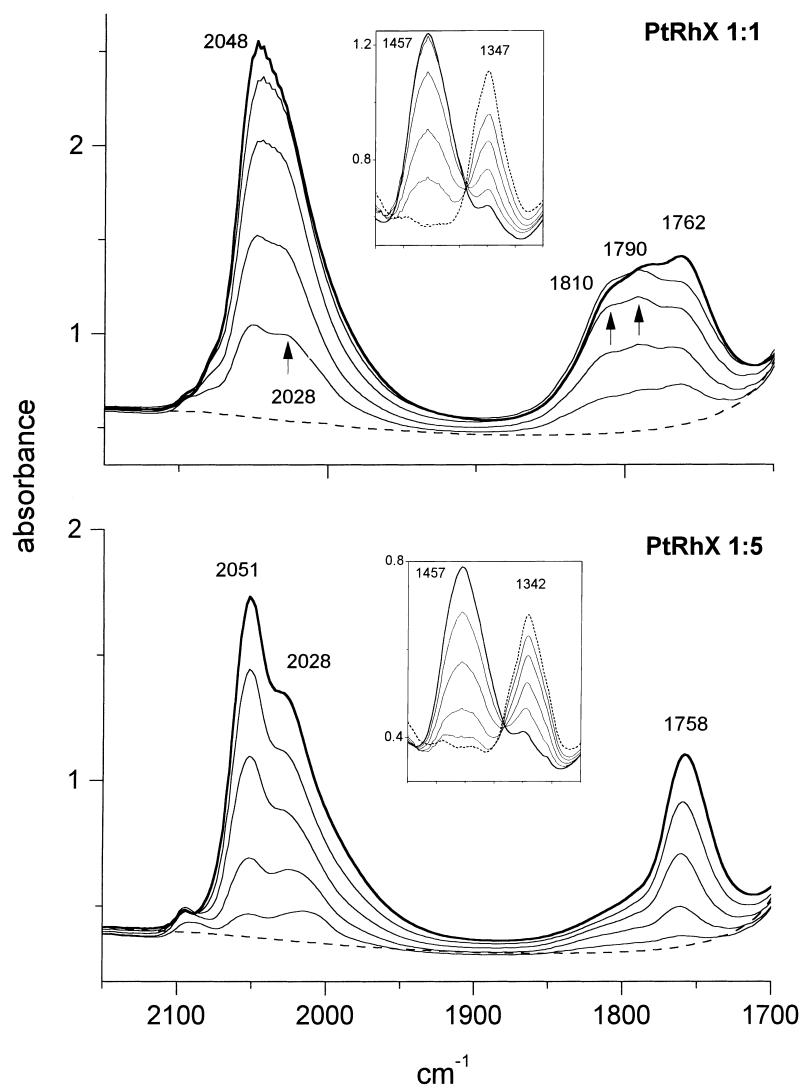


Fig. 2. Carbonylation of PtRhX at 100°C. Top-Pt/Rh 1:1 after: dashed 2 min, solid 0.5, 1, 2 and 4 h, bold solid 15 h. Bottom-Pt/Rh 1:5 after: dashed 2 min, solid 0.5, 1, 2 and 4 h, bold solid 15 h. The same holds for insets.

The carbonylation of PtRhX is shown in Fig. 2. Spectra of Pt/Rh 1:1 (top) exhibit two maxima in the band of bridged CO ligands. The maximum at 1790 cm^{-1} belongs to the orange $[\text{Pt}_3(\text{CO})_6]_2^{2-}$ complex. Position of the other maximum (1762 cm^{-1}) indicates a bimetallic carbonyl: it is just between those of Pt and Rh carbonyls (1778 and 1746 cm^{-1} , respectively, cf. Fig. 1). The carbonylation rate is higher than in cases of PtX and RhX samples.

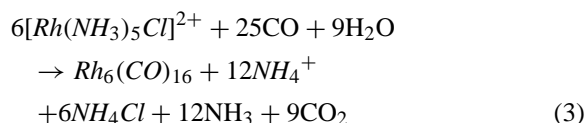
When a sample with twice lower content of both metals was used (Pt/Rh “0.5:0.5”), the carbonylation rate did not decrease which points to an easy migration of Pt and Rh species if the sample is hydrated. Mild dehydration of the parent ammine mixture (1:1) prior to carbonylation hinders the formation of the bimetallic carbonyl, as $\text{Rh}(\text{CO})_2^+$ is formed.

In contrast to the wide band of bridged CO from Pt/Rh 1:1 ratio, the ratio 1:5 enables formation of one

carbonyl species: the band at 1758 cm^{-1} is sharp. The colour of the carbonylated sample is almost white or pale beige. The band of terminal CO at 2051 cm^{-1} is accompanied by a shoulder 2028 cm^{-1} . In the case of mildly dehydrated sample, the band at 1762 cm^{-1} becomes sharp after 60 h when a sufficient amount of $\text{Rh}(\text{CO})_2^+$ agglomerates (not shown).

The carbonylation rate at 100°C is ca by one order of magnitude higher in the first few hours than at 75°C , the carbonylation is almost finished after 6 h. However, only after 36 h the maximum at 1762 cm^{-1} clearly dominates (Fig. 3). The sample was then cooled to 40°C , shortly evacuated and oxidised by 1 atm of O_2 for 30 min (Fig. 3, middle). The decreasing band at 2048 cm^{-1} was shifted to 2057 cm^{-1} , the side bands of the triplet (2092, 2016) belong to $\text{Rh}(\text{CO})_2^+$. The bands of bridged CO decrease more rapidly, being splitted in two bands at 1830 and 1758 cm^{-1} . In order to remove most of terminal CO ligands, the sample was heated from 40 to 100°C for 15 min (bold solid curve). The decreasing band of NH_4^+ at 1460 cm^{-1} partially returns during oxidation to the band of NH_3 ligands at 1335 cm^{-1} (inset). The recarbonylation of oxidised sample proceeded rapidly (Fig. 3, bottom), the spectrum after 3 h is very similar to that of the first carbonylation after 6–36 h. The bands at 2092 and 2016 cm^{-1} , belonging to the intermediate $\text{Rh}(\text{CO})_2^+$, can be due to a little deficit of water caused by preceding evacuation of oxygen at room temperature. The band of NH_4^+ at 1460 cm^{-1} is renewed (inset).

Carbonylation of PtY (Fig. 4, top) differs from that of PtX (Fig. 1, top) in the position of bridged CO (1800 versus 1779 cm^{-1} , respectively). Carbonylated RhY (Fig. 4, bottom) exhibits the well known spectrum of the neutral $\text{Rh}_6(\text{CO})_{16}$ complex with sharp bands at 2098 and 1770 cm^{-1} (1760 cm^{-1} in [11]). The reaction can be described by Eq. (3):



It proceeds very slowly at 100°C (extent ca 15% in 18 h — estimated from the decrease of the NH_3 band at 1335 cm^{-1}). With respect to the slight deficit of NH_3 ligands, intensity of the band of NH_4^+ at 1455 cm^{-1} is substantially higher than in the case of NaX or KY supports where anionic carbonyls are formed [17]. The

reason of this fact is not clear. When the carbonylation extent is higher [17], this discrepancy disappears.

Carbonylation of PtRhY (1:1) is quite complete in 18 h, as follows from Fig. 5, top. The weak band at 2102 cm^{-1} with the transient maximum in 6 h (both PtRh samples) can belong to $\text{Rh}(\text{CO})_2^+$. The 1:1 mixture exhibits a sharp band at 1752 cm^{-1} , analogous to the band 1762 cm^{-1} in NaX. However, this band increases faster than in NaX and dominates also at the beginning of carbonylation, being accompanied by shoulders at 1810 and 1779 cm^{-1} which can be assigned to $[\text{Pt}_3(\text{CO})_6]_2^{2-}$ (the sample is orange).

Addition of an only fivetimes lower Pt amount also affects the carbonylation rate: after 18 h ca 50% of primary complexes reacted to bimetallic carbonyl with CO bands at 2065, 2035 and 1752 cm^{-1} (Fig. 5, bottom).

4. Discussion

The experimental IR spectra of PtRh carbonyls in NaX are compared with the sum of spectra of both monometallic carbonyls (normalised to the wafer thickness and metal amount) in Fig. 6. It follows that the sum of Pt and Rh spectra in 1:1 ratio (top) gives resolved bands of bridge-bonded CO on Pt (1774 cm^{-1}) and Rh (1747 cm^{-1}), while the experimental spectrum exhibits a broad band with a maximum between (1762 cm^{-1}). Also the band of terminal CO's differs from the Pt + Rh sum in the maximum position. The difference is doubtless for the 1:5 ratio (Fig. 6, bottom): in spite of the fivefold Rh excess the band around 2050 cm^{-1} dominates. The sharp band of bridged CO (1758 cm^{-1}) is shifted by 11 cm^{-1} towards the band of sum Pt + 5Rh (1747 cm^{-1}) which corresponds with monometallic Rh carbonyl (cf. Fig. 1, bottom). The experimental IR spectrum points to the formation of a bimetallic PtRh carbonyl, very probably $[\text{PtRh}_5(\text{CO})_{15}]^-$. IR spectra of this species reported in literature show the position of linearly bonded CO between 2034 – 2041 cm^{-1} and rather broad range of bridge-bonded CO: 1785 – 1812 cm^{-1} (Table 1). The spectra found for other PtRh carbonyls exhibit features more different from our experimental values given in Fig. 2 (2–3 bands of terminal CO at lower wave-numbers, cf. Table 1).

Spectra of CO adsorbed on Pt-Rh/ Al_2O_3 were described in [23].

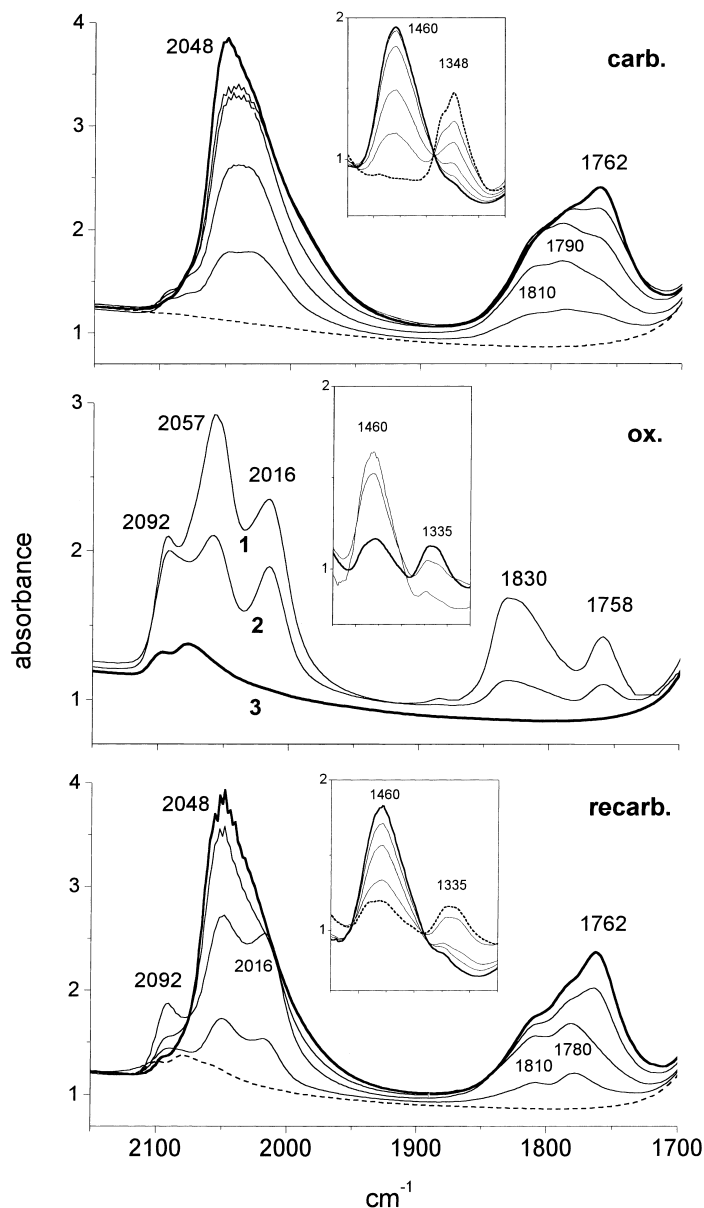


Fig. 3. Carbonylation, oxidation and recarbonylation of PtRh 1:1 in NaX. Top-carbonylation at 100°C after: dashed 2 min, solid 0.5, 1, 2 and 6 h, bold solid 36 h. Middle-oxidation: sp. (1) 2 min at 40°C, sp. (2) 30 min at 40°C; sp. (3) (bold solid) 15 min at 100°C. Bottom-evacuation at 40°C 1 min (dashed sp.) and recarbonylation at 100°C after: 2, 15, 45 min and 3 h (bold solid). The same holds for insets.

Carbonylation of PtRh mixtures in NaY leads to the IR spectra of very similar character as in NaX (cf. Figs. 2 and 5). Experimental spectra of PtRh carbonyls in NaY differ at glance from the sum

of carbonyl spectra of monometallic Pt and Rh, so that this is not displayed. It is very interesting that the carbonylation of monometallic Rh sample does not give polynuclear anionic carbonyls (neither

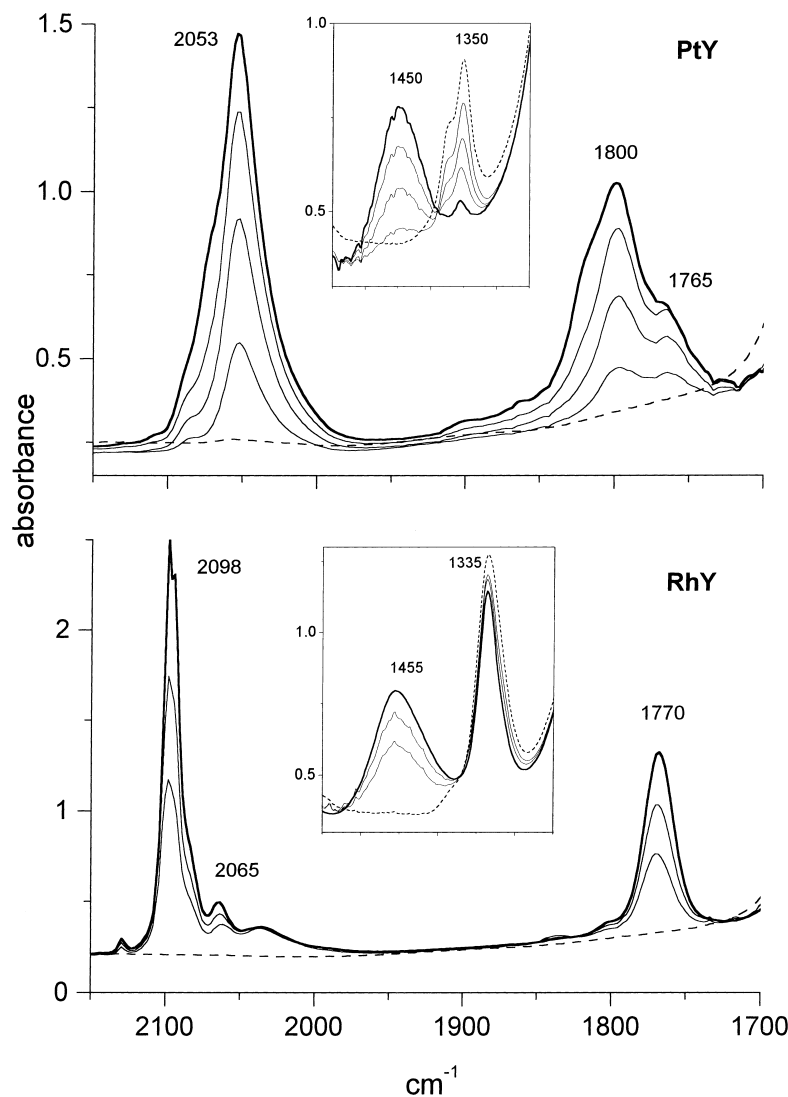


Fig. 4. Carbonylation of PtY and RhY at 100°C. Top-PtY after: dashed 2 min, solid 4, 8 and 12 h, bold solid 18 h. Bottom-RhY after: dashed 2 min, solid 4 and 8 h, bold solid 18 h. The same holds for insets.

$[\text{Rh}_6(\text{CO})_{15}]^{2-}$ nor $[\text{Rh}_5(\text{CO})_{15}]^-$ — only the neutral $\text{Rh}_6(\text{CO})_{16}$ is slowly formed (Fig. 4). Formation of $[\text{Rh}_6(\text{CO})_{15}]^{2-}$ was possible in more basic KY [17]. The fact that $\text{Rh}_6(\text{CO})_{16}$ or $[\text{PtRh}_5(\text{CO})_{15}]^-$ are formed in NaY can be related to the structure of both carbonyls. The neutral $\text{Rh}_6(\text{CO})_{16}$ consists of an Rh octahedron with 12 terminal and four face-bridged CO ligands: $\text{Rh}_6(\text{CO})_{12}(\mu_3\text{-CO})_4$ [24,25]. Except for one terminal CO on Pt, the struc-

ture of bimetallic complex according to [19,20] is the same: $[\text{PtRh}_5(\text{CO})_{11}(\mu_3\text{-CO})_4]^-$.

Monometallic $[\text{Rh}_6(\text{CO})_{15}]^{2-}$ complex with the same numbers of metal atoms and CO ligands was suggested in detail as $[\text{Rh}_6(\text{CO})_9(\mu_2\text{-CO})_3(\mu_3\text{-CO})_3]^{2-}$ [26]. This CO configuration has not been proved for the $[\text{PtRh}_5(\text{CO})_{15}]^-$ complex. Some transient fraction of neutral $\text{Rh}_6(\text{CO})_{16}$ could be expected in NaY, however, the weak band at 2102 cm^{-1} (Fig. 5) rather

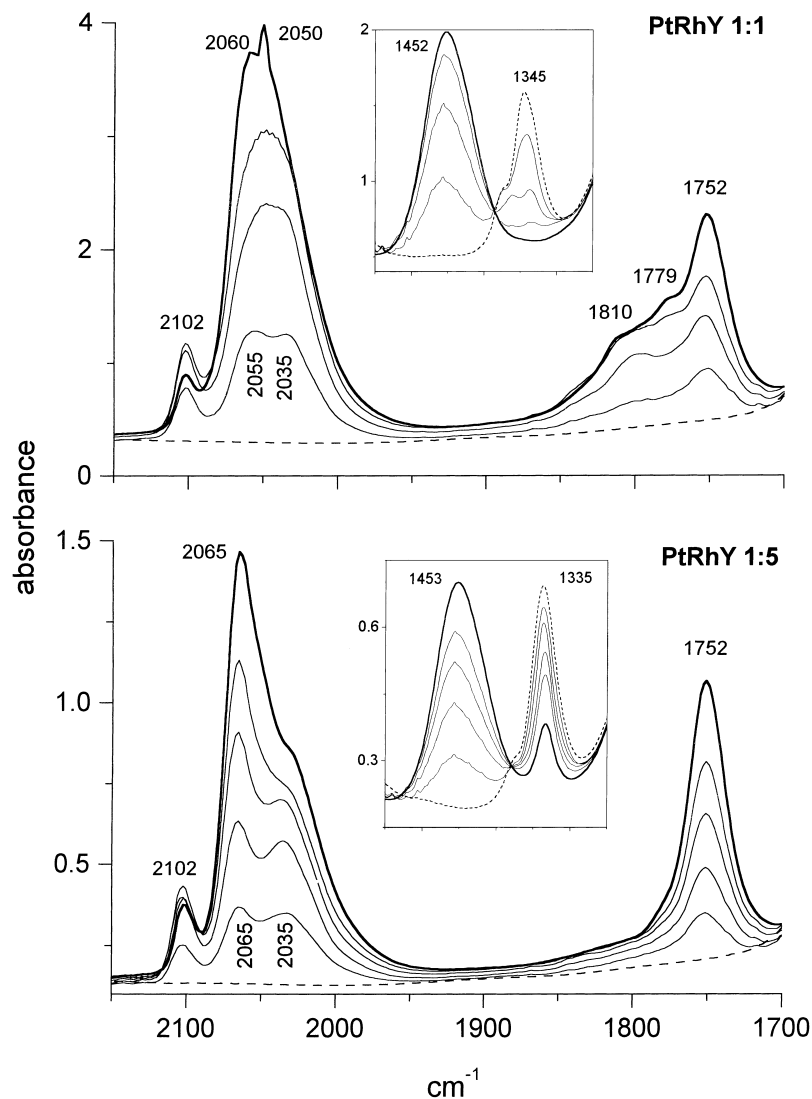
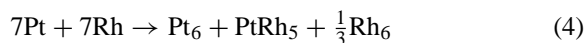


Fig. 5. Carbonylation of PtRhY at 100°C. Top-Pt/Rh 1:1 after: dashed 2 min, solid 4, 8 and 12 h, bold solid 18 h. Bottom-Pt/Rh 1:5 after: dashed 2 min, solid 4, 8, 12 and 18 h, bold solid 41 h. The same holds for insets.

originates from Rh dicarbonyl cation (characteristic bands at 2105 and 2035 cm^{-1} , not shown).

The charge of hexanuclear complexes $[\text{Rh}_6(\text{CO})_{15}]^{2-}$, $[\text{PtRh}_5(\text{CO})_{15}]^-$ and $\text{Rh}_6(\text{CO})_{16}$ correlates with wave-numbers of main terminal CO vibrations (2022, 2053 and 2098 cm^{-1} , respectively). The dependence of wave-number versus charge is roughly linear, in agreement with suggestion reported in [27,28].

If the mixture Pt/Rh 1:1 is carbonylated in NaX, the formation of $[\text{PtRh}_5(\text{CO})_{15}]^-$ together with $[\text{Pt}_3(\text{CO})_6]^{2-}$ proceeds (Fig. 2, top), probably accompanied by low amount of $[\text{Rh}_6(\text{CO})_{15}]^{2-}$ as follows from Eq. (4):



The mixture of products is reflected especially in the broad band of bridge-bonded CO. The mixture Pt/Rh

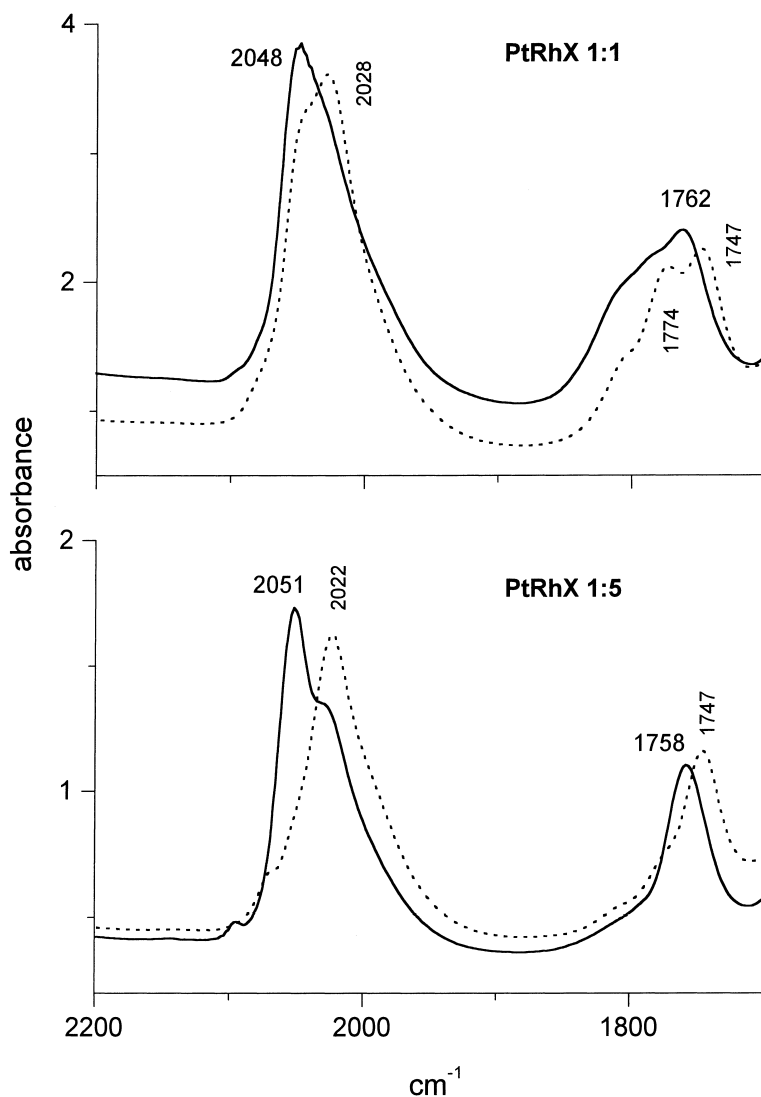


Fig. 6. Comparison of PtRhX with (Pt + Rh)X. Top-Pt/Rh 1:1. Bottom-Pt/Rh 1:5; solid final spectra of PtRhX, dashed normalized sum of PtX + RhX (or 5RhX, corrected) final spectra.

1:5 seems to be the most favourable for the creation of almost pure PtRh₅ carbonyl.

5. Conclusions

Bimetallic anionic carbonyl assigned to [PtRh₅(CO)₁₅]⁻ was prepared in NaX by ship-in-bottle carbonylation of mixed Pt and Rh amines in the

presence of sufficient water amount. The same holds for the carbonylation in NaY although Rh alone reacts much more slowly to only neutral Rh₆(CO)₁₆ complex. PtRh₅ carbonyl can be obtained using various Pt/Rh ratios.

Oxidation and recarbonylation lead rapidly to the original IR spectra of bimetallic carbonyl although the Rh skeleton is decomposed during oxidation.

Acknowledgements

This study was supported by the Grant Agency of the Academy of Sciences of the Czech Republic (A4040710). The author thanks Dr. J. Nováková for comments to the manuscript.

References

- [1] L. Kubelková, J. Vylita, L. Brabec, L. Drozdová, T. Bolom, J. Nováková, G. Schulz-Ekloff, N.I. Jaeger, *J. Chem. Soc., Faraday Trans.* 92 (1996) 2035.
- [2] G.J. Li, T. Fujimoto, A. Fukuoka, M. Ichikawa, *Catal. Lett.* 12 (1992) 171.
- [3] L. Kubelková, L. Drozdová, L. Brabec, J. Nováková, J. Kotrla, P. Hülstede, N.I. Jaeger, G. Schulz-Ekloff, *J. Phys. Chem.* 100 (1996) 15517.
- [4] Z. Bastl, M. Beneke, L. Brabec, N.I. Jaeger, P. Hülstede, J. Nováková, G. Schulz-Ekloff, *Phys. Chem. Chem. Phys.* 2 (2000) 3099.
- [5] M. Beneke, L. Brabec, N. Jaeger, J. Nováková, G. Schulz-Ekloff, *J. Mol. Catal. A: Chem.* 157 (2000) 151.
- [6] L. Drozdová, L. Brabec, J. Nováková, M. Beneke, N. Jaeger, G. Schulz-Ekloff, *Microporous Mesoporous Mater.* 35-36 (2000) 511.
- [7] T. Yamamoto, T. Shido, S. Inagaki, Y. Fukushima, M. Ichikawa, *J. Phys. Chem. B* 102 (1998) 3866.
- [8] M. Sasaki, M. Osada, N. Higashimoto, T. Yamamoto, A. Fukuoka, M. Ichikawa, *J. Mol. Catal. A: Chem.* 141 (1999) 223.
- [9] B.E. Hanson, M.A. Davis, D. Taylor, E.J. Rode, *Inorg. Chem.* 23 (1984) 52.
- [10] E.J. Rode, M.E. Davis, B.E. Hanson, *J. Catal.* 96 (1985) 574.
- [11] L.F. Rao, A. Fukuoka, N. Kosugi, H. Kuroda, M. Ichikawa, *J. Phys. Chem.* 94 (1990) 5317.
- [12] P. Gelin, Y. Ben Taarit, C. Naccache, *J. Catal.* 59 (1979) 357.
- [13] L.F. Rao, S.J. Hwang, T.S. King, M. Pruski, *J. Phys. Chem.* 100 (1996) 5668.
- [14] N. Takahashi, A. Mijin, H. Suematsu, S. Shinohara, H. Matsuoka, *J. Catal.* 117 (1989) 348.
- [15] D.C. Tomczak, V.L. Zholobenko, H. Treviño, G.D. Lei, W.M.H. Sachtler, *Stud. Surf. Sci. Catal.* 84 (1994) 893.
- [16] T.J. Lee, B.C. Gates, *Catal. Lett.* 8 (1991) 15.
- [17] L. Brabec, J. Nováková, *J. Mol. Catal. A: Chem.*, in press.
- [18] A. Fumagalli, S. Martinengo, G. Ciani, N. Masciocchi, A. Sironi, *Inorg. Chem.* 31 (1992) 336.
- [19] A. Fumagalli, S. Martinengo, P. Chini, A. Albinati, S. Bruckner, B.T. Heaton, *J. Chem. Soc., Chem. Commun.* (1978) 195.
- [20] A. Fumagalli, S. Martinengo, P. Chini, D. Galli, B.T. Heaton, R.D. Pergola, *Inorg. Chem.* 23 (1984) 2947.
- [21] Z. Xu, S. Kawi, A.L. Rheingold, B.C. Gates, *Inorg. Chem.* 33 (1994) 4415.
- [22] O.-B. Yang, M. Shirai, W.A. Weber, B.C. Gates, *J. Phys. Chem. B* 102 (1998) 8771.
- [23] J.A. Anderson, C.H. Rochester, *J. Chem. Soc., Faraday Trans.* 85 (1991) 1479.
- [24] E.R. Corey, L.F. Dahl, W. Beck, *J. Am. Chem. Soc.* 85 (1963) 1202.
- [25] L.F. Rao, A. Fukuoka, N. Kosugi, H. Kuroda, M. Ichikawa, *J. Phys. Chem.* 94 (1990) 5317.
- [26] V. Albano, P. Chini, V. Scatturin, *J. Organomet. Chem.* 15 (1968) 423.
- [27] P. Chini, G. Longoni, V.G. Albano, *Adv. Organomet. Chem.* 14 (1976) 285.
- [28] P. Chini, *Rev. Inorg. Chim. Acta.* (1968) 31.