DOI: 10.1002/zaac.202200320

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Journal of Inorganic and General Chemistry

Zeitschrift für

Rhenium Complexes with p-Fluorophenylisocyanide

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p-Fluorophenylisocyanide $(CNPh^{pF})$ reacts with $[Re(CO)_5Br]$ under stepwise exchange of the carbonyl ligands depending on the conditions applied. The reaction stops with the formation of *fac*-[Re(CO)₃Br(CNPh^{pF})₂] in boiling THF. An ongoing carbonyl exchange is observed at higher temperatures, e.g. in refluxing toluene, with the final formation of the $[Re(CNPh^{pF})_6]^+$ cation. The progress of the reactions has been studied by ¹⁹F NMR spectroscopy and the structures of $[Re(CO)Br(CNPh^{pF})_4]$ and $[Re(CNPh^{pF})_6](BPh_4)$ have been elucidated by X-ray diffraction.

The frequent use of an hexakis(isocyanide) complex of technetium (^{99m}Tc-Sestamibi or Cardiolite)^[1-3] in diagnostic nuclear medicine has also stimulated the related chemistry of rhenium.^[4-9] Rhenium complexes are frequently used as (almost) non-radioactive mimics for the corresponding technetium compounds, but there are also two beta-emitting nuclei (¹⁸⁶Re: half-life 90 h, E_{Bmax}= 1.09 MeV and ¹⁸⁸Re: half-life 16.9 h, E_{Bmax}= 2.12 MeV),^[10] which are under discussion for applications in nuclear medical therapy. Particularly ¹⁸⁸Re is readily available for the clinical sites from an alumina-based ¹⁸⁸W/¹⁸⁸Re generator system, from which the isotope can be eluted when it is required.^[11]

Mixed carbonyl/isocyanide complexes are under consideration for a long time not only as potential imaging agents, but also because of their interesting optical properties and as potential anticancer drugs.^[12-16] For both fields, corresponding CO/CNR exchange reactions are of interest and for the most alkyl isocyanides reactions starting from [Re(CO)₅Br] or [Re-(CO)₃(X)₃]^{+,2-} complexes (X = solvent, halide) form products with the *fac*-tricarbonyl core.^[17] Under mild conditions, frequently two isocyanides are introduced and products of the composition [Re(CO)₃(X)(CNR)₂]^{+,0} are formed,^[5,13,17-19] while the introduction of a third isocyanide ligands usually requires a halide scavenger.^[12,18] A slightly different behavior has been observed for aryl isocyanides, which may allow an ongoing

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- Supporting information for this article is available on the WWW under https://doi.org/10.1002/zaac.202200320
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carbonyl exchange depending on their substitutions. First reports about this point can be found in an early paper of Treichel and Williams,^[17] and during our experiments with technetium and rhenium complexes with sterically encumbered m-terphenyl isocyanides we found that particularly the p-fluorinated isocyanide CNp-FAr^{DarF2} (see Scheme 1) does not stop with the replacement of three carbonyl ligands. Irrespective of its steric bulk it forms the tetrakis(isocyanide) complex [Re(CO)Br(CNp-FAr^{DarF2})₄] (3) (Scheme 1).^[20] The tris(isocyanide) complex 2 could be isolated as well for rhenium, while a similar reaction with the common technetium starting material (NBu₄)[Tc₂(CO)₆(Cl₃)] gives quantitatively the tetrakis compound. Such a difference in the reactivity is not unexpected keeping in mind the slower kinetics in exchange reactions of rhenium compared with technetium.^[21]

Very recently, we found that the bulky CNp-FAr^{DarF2} shows a remarkable reactivity also with other synthons such as *fac*-[Tc(CO)₃Cl(CN^tBu)₂] (CN^tBu=t-butylisocyanide), where it replaced selectively the three carbonyl ligands and gave the mixed-isocyanide complex [Tc(CNp-FAr^{DarF2})₃Cl(CN^tBu)₂].^[22] On the basis of DFT calculations,^[22] we address the observed



Scheme 1. Reactions of $[{\rm Re}({\rm CO})_5 Br]$ (1) with the sterically encumbered isocyanide CNp-FAr^{DarF2} $^{[20]}$

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Scheme 2. Stepwise ligand exchange with CNPh^{pF} starting from [Re(CO)₅Br] (1).

reactivity to the fluorine substituent on the central phenyl ring, while the steric bulk of the ligands limits the extend of potential exchange reactions. For a further insight into the electronic and steric effects of such reactions, we performed reactions of $(NBu_4)[Tc_2(CO)_6Cl_3]$ and $[Tc(CO)_3Cl(CN^tBu)_2]$ with p-fluorophenylisocyanide ($CNPh^{pF}$), during which the entire coordination sphere of technetium was replaced and the $[Tc(CNPh^{pF})_6]^+$ cation was formed.^[22] Having in mind the slower ligand exchange kinetics for the 'third transition row' elements, we decided to extend these experiments also to related rhenium complexes.

Attempted reactions of $[\text{Re}(\text{CO})_3\text{Cl}(\text{CN}^t\text{Bu})_2]$ with $\text{CNp}\text{-}\text{FAr}^{\text{Darf}^2}$ did not result in the replacement of carbonyl ligands as was observed for the technetium analog. Even after prolonged heating (3 days!) in toluene there was no evidence for the formation of defined mixed isocyanide complexes and an gradual decomposition of the ligand and intermediate products was observed by subsequently recorded ¹⁹F NMR spectra.

In contrast, reactions with CNPh^{pF} are more defined and the ligand exchange starting from $[\text{Re}(\text{CO})_5\text{Br}]$ proceeds under a stepwise replacement of carbonyl ligands (Scheme 2). The rate of the exchange is generally slow, but can be controlled by the reaction temperature. In boiling THF, up to two CO ligands are replaced. The intermediate formation of the monosubstitution product $[\text{Re}(\text{CO})_4\text{Br}(\text{CNPh}^{\text{PF}})]$ (4) is indicated by a ¹⁹F NMR signal at 109.3 ppm, which could be recorded in the reaction mixture during the early stages of the reaction. Subsequent recordings of ¹⁹F NMR spectra of the reaction mixture are shown in Figure 1, in which only the signals of two ligand exchange products could be detected. While no pure samples of the transient compound 4 could be isolated from such reactions, the final product *fac*-[Re(CO)₃Br(CNPh^{pF})₂] (5) was obtained as a colorless solid in good yields.



Figure 1. ¹⁹F NMR spectra of the stepwise CO/CNPh^{pF} exchange starting from $[Re(CO)_{3}Br]$ (1) in boiling THF and of pure $[Re(CO)_{3}Br-(CNPh^{pF})_{2}]$ (5) in THF.

The IR spectrum of **5** shows the $v_{(CO)}$ stretches at 1916, 1973 and 2026 cm⁻¹, while the $v_{(CN)}$ bands appear at 2153 and 2183 cm⁻¹. The latter values are higher than that of the uncoordinated isocyanide (2129 cm⁻¹), which suggests that the isocyanide does not act as π -acceptor in compound **5**. This is in accord with other rhenium or technetium complexes of the general composition $[M(CO)_3X(CNR)_2]$.^[12,13,17,18,23-28] The ESI + mass spectrum of **5** shows an intense signal at m/z=614.914 for the cluster ion {M+Na}⁺ with the expected isotopic pattern. Details are shown in the Supporting Information.

Prolonged heating of the above mentioned reaction mixture in THF did not give higher carbonyl/isocyanide exchange rates, even when additional Ag(PF₆) was added as Br⁻ scavenger. ¹⁹F NMR spectra of such reaction mixtures indicate a gradual decomposition of CNPh^{pF} in such solutions by the appearances of numerous ¹⁹F signals (see Supporting Information). All our





attempts to isolate the expectes tris complex [Re-(CO)₃(CNPh^{pF})₃]⁺ from such solutions failed.

An ongoing ligand exchange, however, is observed at higher temperatures, e.g. in boiling toluene, and species such as $[Re(CO)Br(CNPh^{pF})_4]$ and $[Re(CNPh^{pF})_6]^+$ could be assigned unambiguously in the ¹⁹F NMR spectra of such solutions. Two more signals with an intensity ratio of 4:1 can be assigned to $[ReBr(CNPh^{pF})_5]$ or $[Re(CO)(CNPh^{pF})_5]^+$. The spectra of such a reaction sequence are shown as Supporting Information. In the course of the reaction, the initially clear mixture became turbid and gradually a colorless solid precipitated. This solid contains charged species (mainly $[Re(CNPh^{pF})_6]Br)$ which are only sparingly soluble in toluene, while the neutral species (mainly $[Re(CO)Br(CNPh^{pF})_4]$ (6)) remain in solution.

Yellow crystals of compound **6** can be obtained by concentration of the mother liquor, filtration from the precipitated salt(s) and crystallization from CH₂Cl₂/n-pentane in a yield of approximately 30%. The compound is readily soluble in polar solvents. The v_(CO) band appears at a low value of 1874 cm⁻¹, but unlike in compound **5** also the v_(CN) frequency (2065 cm⁻¹⁾ is bathochromically shifted against the value in the uncoordinated isocyanide indicating that CNPh^{pF} participates in the withdrawal of electron density from the d⁶ ion by π -backbonding in the tetrakis complex.

The ESI+ mass spectrum of $[Re(CO)Br(CNPh^{pF})_4]$ (6) in acetonitrile shows an intense signal at m/z=740.107, which can be assigned to the $\{M-Br+CH_3CN\}^+$ ion, but also the molecular ion $\{M+Na\}^+$ is visible with lower intensity at m/z=800.986. Similar to the spectrum of compound **5**, also that of $[Re(CO)Br-(CNPh^{pF})_4]$ (6) gives evidence for the formation of dimeric species in the mass spectrometer (for details see Experimental and Supporting Information).

Single crystals of **6** were obtained by slow diffusion of npentane into a solution of the complex in CH_2CI_2 . Figure 2 shows an ellipsoid representation of the molecular structure of the complex with an almost perfectly planar equatorial coordination sphere formed by the four $CNPh^{pF}$ ligands. Minor deviations are only due to the C10-Re–C1/C4 angles being slightly larger than 90° due to the bulk of the CO ligand. Selected bond lengths and angles can found in the Supporting Information.



Figure 2. Molecular structure of $[Re(CO)Br(CNPh^{p})_{q}]$ (6). Ellipsoids represent 50% probability. Hydrogen atoms have been omitted for clarity.



Figure 3. Ellipsoid representation of the $[\text{Re}(\text{CNPh}^{\text{PF}})_6]^+$ cation in **7**. Ellipsoids represent 50% probability. Hydrogen atoms have been omitted for clarity.

The colorless solid removed from the reaction mixture in toluene dissolves in MeOH. Addition of Na(BPh₄) to such a solution gives a colorless precipitate, which is readily soluble in dichloromethane. Single crystals of [Re(CNPh^{pF})₆](BPh₄) were grown from CH₂Cl₂/n-pentane. Since there are hitherto only two examples of homoleptic hexakis(isocyanide)rhenium(I) cations are studied crystallographically, we undertook an X-ray diffraction study on compound **7**.^[9,29] Figure 3 shows the structure of the [Re(CNPh^{pF})₆]⁺ cation. The coordination environment of the rhenium atom is an almost perfect octahedron with *cis*-C–Re–C angels between 87.2(2) and 92.7(2)°. Details about individual bond lengths and angles are contained in the Supporting Information.

The ESI+ mass spectrum of **7** is dominated by the molecular ion at m/z=913.151 and the $v_{(CN)}$ stretch appears at 2074 cm $^{-1}$ in its IR spectrum. The latter value confirms the expected back-donation to the isocyanide ligands in the homoleptic rhenium(I) complex.

Summarizing, it can be stated that the introduction of a fluorine atom in 4-position of the phenyl ring increases the reactivity of arylisocyanides with d⁶ metal centers such as Tc(I) or Re(I) carbonyl complexes and that depending on the reaction conditions applied a complete exchange of the coordination sphere of the metal ions and the formation of $[M(CNPh^{pF})_6]^+$ cations (M = Tc, Re) can be achieved. The exchange rate is faster with technetium, where only the final products could be obtained, while for rhenium intermediates such as $[Re(CO)_3Br-(CNPh^{pF})_2]$ or Re(COBr(CNPh^{pF})_4] could be isolated. The results of the present communication can be regarded as another experimental proof of the old paradigm of Dieter Lentz that "fluorinated isocyanides are more than ligands with unusual properties".^[30]

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Experimental Section

Details about the crystallographic studies and the spectroscopic instrumentation are given as Supporting Information. Unless otherwise stated, reagent-grade starting materials were purchased from commercial sources and either used as received or purified by standard procedures. Solvents were dried and deoxygenated according to standard procedures. The synthesis of CNPh^{pF} was done by adapted literature procedures.^[31]

 $fac-[Re(CO)_{3}Br(CNPh^{pF})_{2}]$ (5): [Re(CO)_{5}Br] (40.6 mg, 0.1 mmol) was dissolved in 4 mL of THF. CNPh^{pF} (120 µL, 1 mmol) was added and the mixture was heated on reflux for 8 h. The progress of the reaction was monitored by ¹⁹F NMR spectroscopy. After completion of the reaction, all volatiles were removed and the obtained light brown residue was suspended in *n*-hexane, filtered and washed with several portions of *n*-hexane. The obtained colorless solid was dried under reduced pressure. Yield: 45 mg (0.076 mmol, 76%). Elemental analysis: Calcd. for: C17H8BrF2N2O3Re: C, 34.47, H, 1.36, N, 4.73, found: C, 34.90, H 1.62, N, 4.78. IR (ATR, cm⁻¹): ν(CN) 2188 (sh), v(CN) 2153 (s), v(CO) 2026 (s). v(CO) 1973 (s), v(CO) 1916 (s). ¹H NMR (CD₂Cl₂, ppm): 7.53 (m_c, 4H), 7.18 (m_c, 4H). ¹³C{¹H} NMR (CD₂Cl₂, ppm): 186.6, 184.2, 163.4 (d, ¹J(¹⁹F-¹³C) = 250 Hz), 143.9, 129.7 (d, $^{3}J(^{19}F^{-13}C) = 9$ Hz), 123.4, 117.5 (d, $^{2}J(^{19}F^{-13}C) = 24$ Hz). ^{19}F NMR (CD₂Cl₂, ppm): -107.48 (m_c). ESI + MS (acetonitrile): 614.914 ([M+ Na]⁺; calcd 614.914), 630.887 ([M+K]⁺; calcd 630.888), 1206.835 ([2 M+Na]⁺; calcd 1206.838).

 $trans-[Re(CO)Br(CNPh^{pF})_4]$ (6): [Re(CO)₅Br] (40.6 mg, 0.1 mmol) was dissolved in 6 mL of toluene. CNPh^{pF} (120 µL, 1 mmol) was added and the reaction mixture was heated on reflux for 4 h. Another portion of $CNPh^{pF}$ (120 μ L, 1 mmol) was added and the mixture was heated on reflux for another 14 h. The volatiles were removed in vacuum, methanol (5 mL) was added and the remaining solid was filtered off. It was washed with methanol (2 mL) and dissolved in CH₂Cl₂. Diffusion of n-pentane into this solution gave yellow crystals suitable for X-Ray diffraction. They were isolated by filtration and washed with MeOH (2 mL) and n-pentane (2 mL) and dried under reduced pressure. Yield: 24 mg (0.031 mmol, 31%). Elemental analysis: Calcd. for: C₂₉H₁₆BrF₄N₄ORe: C, 44.74; H, 2.07; N, 7.20. Found: C, 44.87; H, 2.40; N, 6.79. IR (ATR): v(CN) 2065 (s), v(CO)1874 (s), ν(CO) 2040 (s). ¹H NMR (CD₂Cl₂, ppm): 7.43 (m_c, 8H, m, aryl-H), 7.12 (m_c, 8H, aryl-H). ¹³C{¹H} NMR (CD₂Cl₂, ppm): 190.71, 162.39 (d, $^{1}J(^{19}F^{-13}C) = 250$ Hz), 154.63, 129.02 (d, $^{3}J(^{19}F^{-13}C) = 9$ Hz), 125.40, 117.07 (d, ${}^{2}J({}^{19}F-{}^{13}C) = 24$ Hz). ${}^{19}F$ NMR (CDCl₃, ppm): -110.7 (m_c). ESI + MS (acetonitrile): 740.108 ([M–Br+MeCN]⁺; calcd 740.108), 800.986 ([M + Na]⁺; calcd 800.990), 1477.076 ([2M-Br]⁺; calcd 1477.082, 1578.985 ([2 M + Na]; calcd 1578.990), 1595.957 ([2 M + K]; calcd 1594.964).

 $[Re(CNPh^{pF})_{a}](BPh_{a})$ (7): Na(BPh_{a}) (340 mg, 1.00 mmol) was added to the filtrate obtained from the synthesis of compound 6, which resulted in the precipitation of a colorless solid. The solid was filtered off and was washed with methanol (3 mL) and diethyl ether (3 mL). Recrystallization was performed from CH₂Cl₂/pentane. Colorless crystals suitable for X-diffraction were obtained. The crystals were washed with MeOH and diethyl ether and dried under reduced pressure. Yield: 37 mg (0.030 mmol, 30%). Elemental analysis: Calcd. for: C₆₆H₄₄BF₆N₆Re: C, 64.34; H, 3.60; N, 6.86. Found: C, 63.36; H, 3.48; N, 6.25. IR (ATR, cm $^{-1}$): $\nu(CN)$ 2074 (s). 1H NMR (CD₂Cl₂, ppm): 7.44–7.35 (12H, m, pF-aryl-H), 7.30 (8H, dt, J=7.1, 3.4 Hz, phenyl-H), 7.19-7.09 (12H, m, pF-aryl-H), 7.02 (8H, t, J= 7.3 Hz, phenyl-H), 6.86 (4H, t, J=7.2 Hz, phenyl-H). $^{13}\text{C}\{^1\text{H}\}$ NMR (CD_2CI_2, ppm) : 164.61 (q, ${}^{1}J({}^{11}B{}^{-13}C) = 50 Hz$), 162.59 (d, ${}^{1}J({}^{19}F{}^{-13}C) =$ 250 Hz), 136.48 (s); 128.89 (d, ³J(¹⁹F-¹³C) = 9 Hz), 126.17 (g, ²J(¹¹B-¹³C) -= 3 Hz), 125.11, 122.24, 117.48 ${}^{2}J({}^{19}F-{}^{13}C) = 25$ Hz), the isocyanide carbon was not observed, probably due to line broadening. ¹⁹F NMR (CD₂Cl₂, ppm): -109.54 (m_c). ESI + MS (acetonitrile): 913.151 ([M + H]⁺, calcd 913.152).

Acknowledgements

We gratefully acknowledge financial support from the DFG (Deutsche Forschungsgemeinschaft: Graduate School BIOQIC), the U.S. National Science Foundation (International Supplement to CHE-1802646) and the Alexander von Humboldt Foundation (Fellowship to J.S.F.). We also acknowledge the assistance of the Core Facility BioSupraMol supported by the DFG. Open Access funding enabled and organized by Projekt DEAL.

Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available in the supplementary material of this article.

Keywords:	Rhenium	•	Isocyanides	•	Carbonyls	•	X-Ray
Crystallography • Ligand Exchange							

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Manuscript received: September 27, 2022 Accepted manuscript online: October 10, 2022