Synthesis, Characterization and Catalytic Applications of $NiCO_4$

M.Sc. Dissertation
by

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DISSERTATION

Submitted in partial fulfilment of
The degree of M.Sc. (Inorganic Chemistry)

By
Mr. Rohidas D. Parab

To
School of Chemical Sciences
Goa University
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DECLARATION

I hereby declare that the work embodied in this report entitled **"Synthesis, Characterization and Catalytic Applications of $NiCO_4$"** was carried out by me during the year 2021-2022 under the guidance of Dr. Kedar Umakant Narvekar. In keeping with the general practice of reporting scientific observations, due acknowledgements have been made wherever the work described is based on the findings of other investigators.

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Rohidas D. Parab

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Synthesis, Characterization and Catalytic Applications of $NiCO_4$

INTRODUCTION

One of the most active areas of research at present time is the study of metal complexes.carbonmonoxide groups are ligands.Indeed,during the last two decades preparative and structural work involving metal carbonyl compounds has provided as great stimulus for the development of inorganic chemistry as have the great advances in boron hydride chemistry,fluorine chemistry or the chemistry of so called pi complexes.In the case of the latter,progress has been intimately connected with the work on the carbonyl complexes.Schutzenberger first recognised carbonmonoxide as a ligandin his preparation and characterizationof $Pt(CO)_2Cl_2$,but the first binary metal carbonyl,tetracarbonylnickel was discovered in 1890.

Some early studies such as that which established the tetrahedral configurationof $Ni(CO)_4$,as well as Sidgwicks and Baileys concept relating the formula of the known metalcarbonyl to an effective atomic number rule were of fundamental importance.It was during this period that W.Hieber and his students made the first of their many significant experimental contributions,including the synthesis of several metalcarbonyls.

Nickeltetracarbonyl is an organonickel compound with the formula $Ni(CO)_4$.It is colourless liquid having very high toxicity and rapid skin absorption.Nickeltetracarbonyl was an intermediate in monds process for producing very high purity nickel but this process is out of use because of its hazardous nature.

Some properties of $Ni(CO)_4$	
Chemical formula	$Ni(CO)_4$
Molar mass	170.73 g/mol
Density	1.319 g/cm^3
Melting point	-17.2 deg C
Boiling point	43 deg C
Solubility	Miscible in most organic solvents

Structure	
Coordination geometry	Tetrahedral
Molecular shape	Tetrahedral
Dipole moment	Zero

LITERATURE REVIEW

Nickeltetracarbonyl formation on non equilibrium Ni surfaces

V.K Medvedev, R.Borner, N.Kruse studied the dissolution process leading to the formation of Nicketetracarbonyl,i.e Ni+4CO→$Ni(CO)_{4(g)}$.The reaction was favoured by high CO gas pressures and equilibrium thermodynamic data (k=7.2*103 bar^{-3} corresponding to DG^0 = -22.6 Kj mol^{-1}) which predict insignificant $Ni(CO)_4$ formation at low pressures. However, deviations of Ni surface topography from its eqlb form may be suspected to alter the kinetics of reaction. Kinetic studies of sub carbonyl formation demonstrated that synthesis dominates over CO decomposition as long as the compression structure of chemisorbed CO is formed sufficiently fast.

In this experiment a standard stainless steel chamber along with an auger electron spectrometer was used .The base pressure in this chamber was 10^{-10} mbar. The rate of $Ni(CO)_4$ formation remained below the detection limits of quadrapole mass spectrometer therefore they adopted a chemical trapping technique by making use of an auxiliary substrate[Rh(110) single crystal] onto which $Ni(CO)_4$ molecules were precipitated in the form of Ni atoms and analysed in auger study.In a first stage of experimental procedure a non equilibrium Ni surface with high step site density was created by sputerring at room temperature. During the reaction experiments,the Rh crystal was held at constant temperature of 480 K in order to ensure low CO coverages and high $Ni(CO_4)$ dissociative sticking probabilities. Ni foil used in this experiment was electropolished in a concentrated solution of HCl.

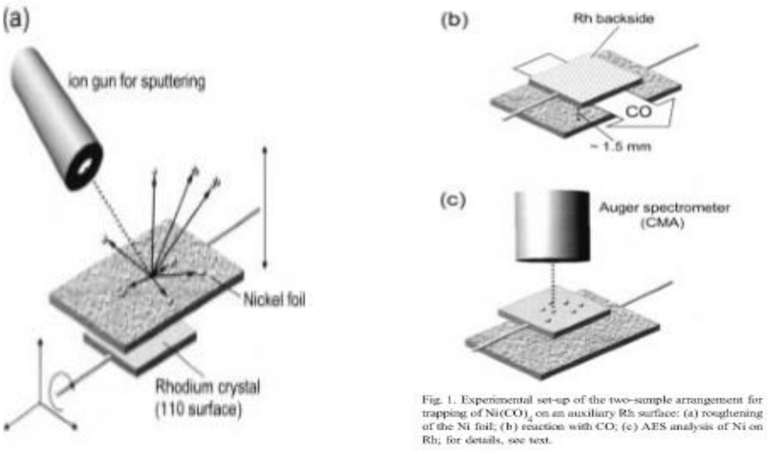


Fig. 1. Experimental set-up of the two-sample arrangement for trapping of $Ni(CO)_4$ on an auxiliary Rh surface: (a) roughening of the Ni foil; (b) reaction with CO; (c) AES analysis of Ni on Rh; for details, see text.

(Medvedev et al., 1998)

Formation of $Ni(CO)_4$ during the interaction between CO and silica supported nickel catalyst.

M.Mihaylov,K.Hadjiinov and H.Knozinger studied the formation of $Ni(CO)_4$ by the interaction of CO with silica supported nickel metal by FTIR.In this experiment carbon monoxide participates as a product or reagent so the knowledge of interaction between CO and Ni was needed.Investigations had shown that CO corresively interacts with metallic Ni which can cause both morphological changes of metal paricles and nickel loss.The formation of $Ni(CO)_4$ is thermodynamically favoured by high pressures and low temperatures.Studies of interaction between Ni^0/SiO_2 and CO at 85k was carried out since there were kinetic reasons for formation of $Ni(CO)_4$ at low temperatures.

In this experiment silica was taken as a support which was commercially available aerosil with specific surface area of $336m^2g^{-1}$.Grafting of Ni+2 ions on the support was performed from 0.1M Ni^{+2} aqueous solution obtained from $Ni(NO_3)_2$ containing 12.5 % ammonia. 10g of SiO_2 were dispersed in 150ml of this solution and the suspension obtained was stirred for 1hr.The precipitate was then filtered thoroughly with water,dried and calcined for 1 hr at 623 K.The IR spectra was recorded on a bruker IFS66 apparatus at spectral resolution of of 1 cm^{-1} accumulating 128 scans. The design of the cell permitted IR measurements in the range between 85k and room temperature. Before the experiments the sample was activated by successive thermooxidative and thermovaccum treatment. The sample was then reduced for 1 hr in hydrogen flow.

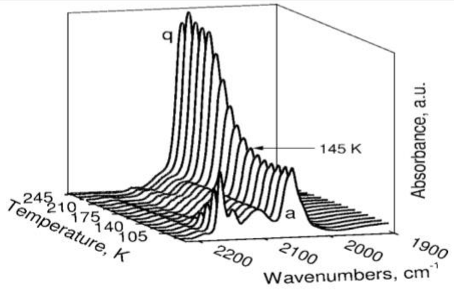


Figure 1. IR spectra of CO (200 Pa equilibrium pressure) adsorbed on Ni^0/SiO_2 at 85 K (a) and at gradually increasing temperature by steps of 10 K up to 245 K (b)–(q).

(Mihaylov et al., 2001)

Jet cooled FTIR spectroscopy and analysis of the µ5 CO stretch fundamental of $Ni(CO)_4$

P.Asselin,P.Soulfard and V.Boudon recorded the revibrational spectra of CO stretch band of $Ni(CO)_4$ at $0.006cm^{-1}$ resolution by coupling a low pressure continuous supersonic jet with a high resolution interferometer.For analysis electron diffraction value for the ground state rovibrational constantβ_0 was used and the excited state rovibrational constant was fixed at order 3.

In this experiment supersonic jet FTIR spectrometer was set up.$Ni(CO)_4$ obtained from stream was seeded in argon at about 20% by bubbling the rare gas through the liquid metal carbonyl into a constantly cooled bath maintained at about 60^0c. The 1 mol h1 flow rate of the $Ni(CO)_4$ = Ar gas mixtures expanded at a stagnation pressure of 80 mbar through a 0.74 mm circular nozzle into a vacuum chamber evacuated at a background pressure of 103 mbar by a 18 m3 h1 Varian four stage oil diffusion pump backed by a 400 m3 h1 roots pump (Edwards 500 H) and a 60 m3 h1 rotary vane pump (Leybold D60). The supersonic expansion is finally probed by the 16-pass arrangement of the IR beam issued from a Bruker IFS 120 HR interferometer and then focused on a InSb detector equipped with anarrow bandpass filter centred around the C–O stretching region (1950–2100 cm^{-1}). A typical jet spectrum of the µ5 stretching band of $Ni(CO)_4$ represents the Fourier transform of 10 coadded interferograms recorded at 0.006 cm^{-1} $Ni(CO)_4$ possesses nine normal modes of vibration; two non-degenerate modes with A1 symmetry (µ1and2), two doubly-degenerate modes with E symmetry (µ3 and 4), one triply-degenerate mode with F1 symmetry (µ9) and four triply-degeneratemodes with F2 symmetry (µ5, 6, 7 and 8). Only F2 fundamentals are infrared active. 5 corresponds to a C–O stretch mode. Since both the 12C and 16O atoms have a zero nuclear spin, the spin statistical weights of $Ni(12C\ 16O)_4$ are such that only levels with A1 or A2 rovibrational symmetry species are allowed (in this work, they only considered this main isotopologue, the others not being observed under the present experimental conditions).

(Asselin et al., 2008)

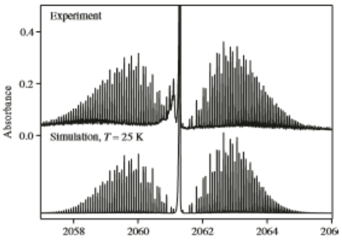


Figure 2. Overview of the spectrum of the ν_5 band of $Ni(^{12}C^{16}O)_4$, compared to the simulation.

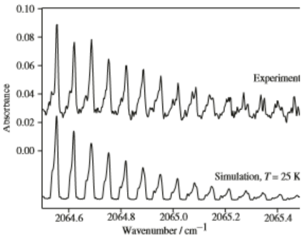


Figure 3. Detail in the R branch of the ν_5 band of $Ni(^{12}C^{16}O)_4$, compared to the simulation.

A Mass Spectrometric Investigation of Nickel Tetracarbonyl and Iron Pentacarbonyl

Robert winters and Robert kiser investigated mass spectrometer of $Ni(CO)_4$ and iron pentacarbonyl. Various metal carbonyl compounds have been found to have excellent catalytic and complexing characteristics.. Therefore, they had undertaken investigations of these carbonyls using mass spectrometric techniques. Because of the interest in these carbonyl compounds, they had commenced mass spectrometric study of a number of transition metal carbonyl compounds by examining the carbonyls of two group VI11 elements, iron and nickel. From the experimental data, the second ionization potentials of $Ni(C0)_4$ and FeCO are calculated. The observation is made that the dissociation processes subsequent to ionization apparently involve successive losses of CO groups.

In this experiment Mass spectra for each of the compounds were obtained at nominal electron energies of 70 e.v. Appearance potentials were determined using the method of extrapolated voltage differences, described by Warren. Ionization efficiency curves were plotted for each determination and the linear portions of the curves forced parallel. The voltage differences at given currents were plotted as a function of the current, and the value of AB obtained upon extrapolation to zero current was added algebraically to the ionization potential of the calibration gas employed. Xenon mixed with the compound being investigated was used to calibrate the ionizing voltage. The known spectroscopic value (12.13 e.v.) for the ionization potential of xenon was used. The heats of formation of both transition metal carbonyls were determined by Fischer and cotton. The values for nickel tetracarbonyl~ and iron pentacarbonyl are -1145.1 and -173.7 kcal/mole, respectively. The mass spectra of the two transition metal carbonyls exhibit many similar characteristics. The most abundant species are the metal ion and the rnonocarbonylmetal ion. However, in the mass spectrum of nickel tetracarbonyl, the dominant species is the NiCO+ and in the iron penta- carbonyl spectrum, the Fe+ is of greatest intensity.

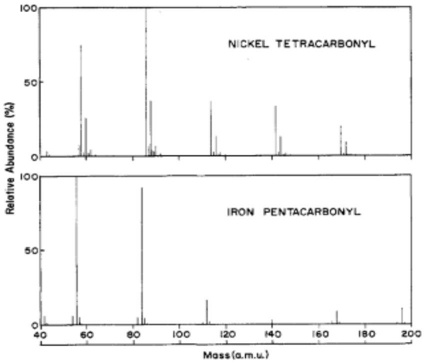


Fig. 1.—Mass spectral cracking patterns of nickel tetracarbonyl and iron pentacarbonyl.

(Winters & Kiser, 1964)

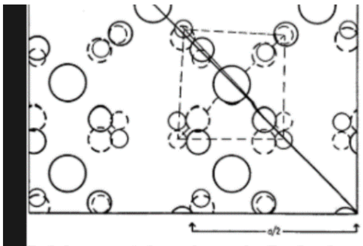
The Crystal Structure of Nickel Carbonyl, Ni(CO)4

Joshua ladell,Benjamin post and Fankuchen determined Single-crystal oscillation diagrams at -55 deg.C These have been indexed on the basis of a cubic unit cell with a 0 ---- 10-84±0.02 . There are 8 molecules per unit cell. The most probable space group is Pa3. Electron-density projections indicate that the configuration of the molecule is that of a regular tetrahedron. The analysis of one- and two-dimensional sections of three-dimensional Fourier syntheses indicates the following bond lengths: :Ni-C ~- 1.84±0.03 A, and Ni-O -~ 2.99±0-03 /k. These are in good agreement with known values determined by means of electron diffraction. The calculation of the electron density along a cube diagonal indicates that the atomic arrangement is Ni-C-O and not the alternative possibility Ni-O-C.

Nickel carbonyl is one of a large group of relatively simple compounds, because they happen to be liquids or gases at room temperature, have not been investigated by X-ray diffraction methods. This paper reports the results of an X-ray diffraction investigation of crystalline nickel carbonyl. These furnish independent determinations of molecular configuration and bond lengths. They show that the carbon, and not the oxygen, atoms are adjacent to the nickel atoms.

Specimens were prepared for X-ray use by vacuum distillation of small quantities of nickel carbonyl into thin-walled pyrex glass capillaries from 0.2 to 0.5 mm. inside diameter. The low-temperature single-crystal oscillation camera used in this investigation has been described elsewhere. A powder camera, 71.6 ram. in radius, was also adapted to low-temperature studies and was used for the determination of lattice constants. Optical examination of single crystals from -- 150 ° C to --25 ° C. gave no indication of solid-phase transitions in this temperature range. Except for one powder diagram obtained at --100°±5 ° C., all powder and single-crystal diagrams were obtained at -550±5 ° C. A measure of the coefficient of expansion of the solid is furnished by differences in spacings between the --55 ° C. and --100 ° C.

Unfiltered copper radiation was used, the nickel atoms in the specimen acting as a filter. Intensities of single-crystal reflections were estimated visually, using multiple-film techniques (Robertson, 1943). The usual Lorentz and polarization corrections were applied. The single crystals studied were cylindrical in shape and their diameters were less than half the optimum thickness; absorption corrections were there- fore considered unnecessary. In computing structure factors, the atomic scattering factor of nickel was corrected for the change in scattering power caused by dispersion by K electrons.



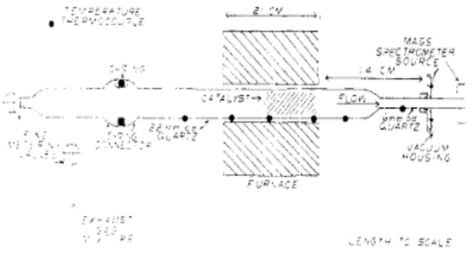
ig. 3. Arrangement of atoms in one unit cell projected on cube face. Origin is at center of drawing. The quadrilateral indicates one molecule in projection.

(Ladell et al., 1952)

Mass Spectrometry of Nickel Carbonyl for Modeling of Automobile Catalysts

J.E Campana and Risbey investigated charge exchange mass spectrum of nickel carbonyl has been reported using a typical gasoline engine exhaust gas as the reactant gas. The effect of pressure on the intensity of both the reactant gas ions and the nickel carbonyl was studied in an attempt to find the optimum condition for the quantification of nickel carbonyl. Using these data, the minimum detectable limit was found to be 10 ppb for nickel carbonyl. This methodology was used to monitor nickel carbonyl in the ef- fluent from a model reactor for a catalytic controlled auto- mobile. Based on these studies, no measureable quantities of nickel carbonyl can be expected from catalytic controlled automobile.

Nickel carbonyl [nickel tetracarbonyl, Ni(CO)4] is one of the most dangerous chemicals known. It exhibits acute toxicity, carcinogenicity, teratogenicity, and can be produced spontaneously in unsuspecting environments whenever carbon monoxide. The Occupational Safety and Health Administration has set this level at 0.007 mg/m3 or 1 ppb (I), which has dictated a need for ultratrace methods of analysis for nickel carbonyl in ambient air. Recently the instrumental methods, infrared spectrophotometry (IR) (21, Fourier transform IR (FTIR) (3,4), plasma chromatography (4), and chemiluminescence (5), have been used to analyze for nickel carbonyl. All of these methods have the advantage of direct measurement with detection limits of about 1 ppb and are also adaptable to process stream monitoring. The methods based on the FTIR and the plasma chromatography methods have been compared in real sample analyses and agree within a few percent. The chemilumi- nescent analysis for nickel carbonyl demonstrates a detection limit of 0.01 ppb with a linearity over four orders of magnitude. Therefore, it is suitable for industrial applications from both the practical and economic stand point. Each daughter ion formed by the successive removal of a neutral CO group "peaked-out" at a successively higher electron energy .This was typical of all the metal carbonyls that were studied and suggested that the fragmentation of metal carbonyls occurred by successive re- moval of neutral CO groups. Pyrolysis and molecular beam studies of nickel carbonyl have shown that the relative intensities of the species in a nickel carbonyl spectrum vary about 10% over the temperature range of 19 to 200 "C (11) and the absolute intensities decrease by about 9670 in that temperature interval. The analysis was performed using a chemical ionization mass spectrometer (Scientific Research Instxuments Corporation, Biospect System) which has been described previously (20). The reactant gas used was a mixture typical of' an automobile exhaust gas. The mass spectrometer source housing pressure was measured with a Bayard-Alpert ionization gauge, but no direct pressure measurement was made in the mass spectrometer source. All the reactant gas was derived from the sample preparation. The composition of the reactant gas (exhaust-gas mixture), nitrogen, carbon monoxide, carbon dioxide, and oxygen, will produce charge exchange (CE) reactions at high source pressure.



(Campana & Risby, 1980)

Nickel-Catalysed Reactions of Allyl Halides and Related Compounds

G. P. Chiusoli and L.Cassarix studied Allyl compounds which react with carbon monoxide, or with carbon monoxide and acetylene, in the presence of tetracarbonylnickel as catalyst. These catalysts are involved also in coupling reactions and addition reactions of activated olefins and ketones. Reactions of this type can be used for the preparation of unsaturated aliphatic acids, esters, nitriles, hydrocarbons, alicyclic ketones, keto acids, keto esters, diketones, lactones, phenols, and other compounds. The reactions proceed via π-allylnickel complexes.

In this experiment new method which we have developed, and in which finely divided nickel and thiourea or iron powder, nickel chloride, and thiourea is used as catalyst, seems even more advantageous With these systems the reaction can be carried out at or even below room tempera- ture (-30 “C with Raney nickel/thiourea) and at atmos- pheric pressure in the absence of tetracarbonylnickel. This new method is useful for the preparation of esters according to equation (b); on the other hand, tetra- carbonylnickel is recommended for the synthesis of acids (equation (b) where R = H). Tetracarbonylnickel can be conveniently prepared from carbon monoxide, iron powder, nickel chloride, and sulfides in water.

$$\mathbf{Fe + NiC1_2 + 4\,CO + Ni(CO)_4 + FeCl_2}$$

Other aliphatic halides, and even benzyl chloride, do not react under the very mild conditions used for reactions with tetracarbonylnickel. However, at temperatures above 100 OC, iodobenzene and other aromatic iodine derivatives react with tetracarbonylnickel and acetylene to form y-keto acids or their esters[O], which may be

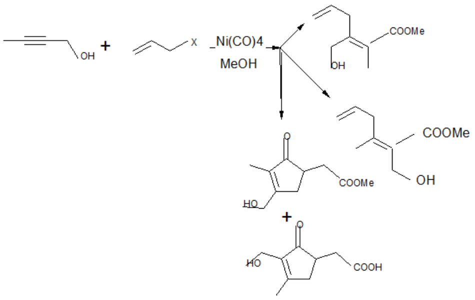
$$\mathbf{C_{,5}H_5 + HCCH + Ni(C0)_4 + 2H_2O + C_6H_5\text{-}CO\text{-}CH_2\text{-}CH_2\text{-}COOH + NiOH + 2\,CO}$$

regarded as hydrolysis products of P,y-unsaturated y-lactones or as hydrogenation products of a,P-unsatu- rated y-keto acids or esters. The a$-unsaturated y-keto acids or esters are hydrolysed under the conditions of the reaction, but owing to the presence of electron- attracting substituents, they do not take up CO.

When a halide of an organic acid reacts with carbon monoxide, acetylene, and tetracarbonylnickel in the presence of an a$-unsaturated aldehyde, the products obtained are esters of oxocyclopentenylidenemethanol or of oxocyclopentenylmethanol, depending on whether hydrogen is given off or taken up.

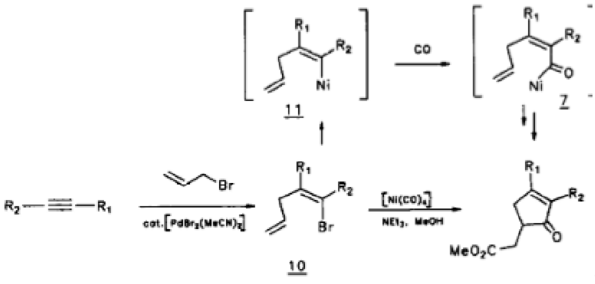
The [Ni(CO)$_4$ promoted carbonylative cycloaddition of alkynes and ally1 halides for the synthesis of 2cyclopentenone derivat ives.

Amadeu Llebaria and Josep Moreto studied [Ni(CO)$_4$]-mediated carbonylative cycloaddition of alkynes and allyl halides. After several unsuccessful attempts to prepare 2 starting from ally1 bromide and 2-butynol [2], they applied other butenolide syntheses to the reagents they had in hand. They found one reported by Chiusoli et al. using an alkynol and an ally1 halide, where cyclization was accomplished in one step, the lactone carbonyl group being incorporated using (Ni(CO)$_4$. After performing the reaction they were surprised to find that a mixture of two regioisomeric cyclopentenones 4 and 5 had been formed instead of the desired lactone. Certainly, cyclopentenones were already reported to arise from this reaction, sometimes as major products, but rarely reaching the 60% yield obtained for this particular alkyne. Considering these results, the practically simultaneous formation of four C-C bonds, and the high regiocontrol displayed for a disubstituted alkyne, we endeavoured to find all the factors influencing the outcome of this reaction for further synthetic applications. The mild reaction condi- tions and use of a common solvent were further advan- tages that outweighted the hazardous use of nickel carbonyl.



(Llebaria & Moretó, 1993)

Simultaneous presence of anyfree monosubstituted alkyne and acyl-Ni intermediates 8 or 9 was detrimental for formation of a discrete cyclopentenone due to further alkyne insertion. How- ever, we succeeded in performing the ally1 insertion separately by means of the catalytic presence of PdII leading to the intermediate bromodiene 10 and then we accomplished the remaining steps with a new [Ni(CO),]-based system .The regioselectivity was in the same sense and even more strict than that of the conventional reaction, allowing the corre- sponding cyclopentenones to be obtained in moderate-to-good yields.



(Llebaria & Moretó, 1993)

CONCLUSION

1. In first article,since both CO chemisorption as well as $Ni(CO)_3$ intermediate and $Ni(CO)_4$ product formation seem to be faster,$Ni(CO)_2$ disruption from kink site is determined as rate determining step.
2. In second article at temperatures below 145 K the interaction between CO and Ni^0/SiO_2 is restricted to the formation of nickel car- bonyls, among them very minor amounts of $Ni(CO)_4$ adsorbed on SiO_2.
3. In third article work constitutes the first detailed analysis of the v5 C–O stretch fundamental of $Ni(CO)_4$, including the Coriolis constant and non-scalar terms that induce tetrahedral splitting.
4. In the fourth article the fragmentation of these transition metal carbonyl ions occurs by successive removal of neutral CO.
5. In the fifth article results of this investigation confirm the tetrahedral configuration and the bond lengths reported by Brockway & Cross (1935) on the basis of electron-diffraction measurements..
6. In the sixth article ion $[Ni(CO)]^+$. (curve 1) was initially the most intense ion and decreased pseudo-
7. In the seventh article catalytic reactions of Ni(CO)4 synthesised using new method were used for many allyl compounds reacting with carbonmonoxide.
8. In the eighth article carbonylative cycloaddition of acetylenes and allylic halides mediated by $[Ni(CO)_4]$ represents a straightforward method for the synthesis of 2-cyclopentenone derivatives.

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