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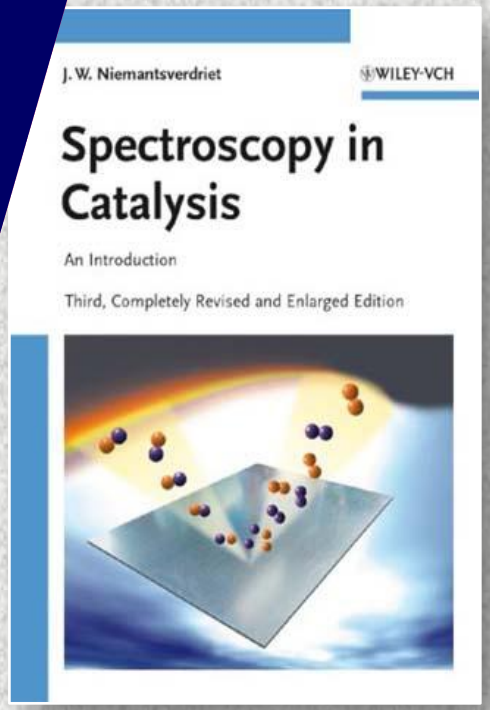
Characterization of solid catalysts

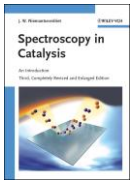
1. Introduction



Prof dr J W (Hans) Niemantsverdriet
Schuit Institute of Catalysis

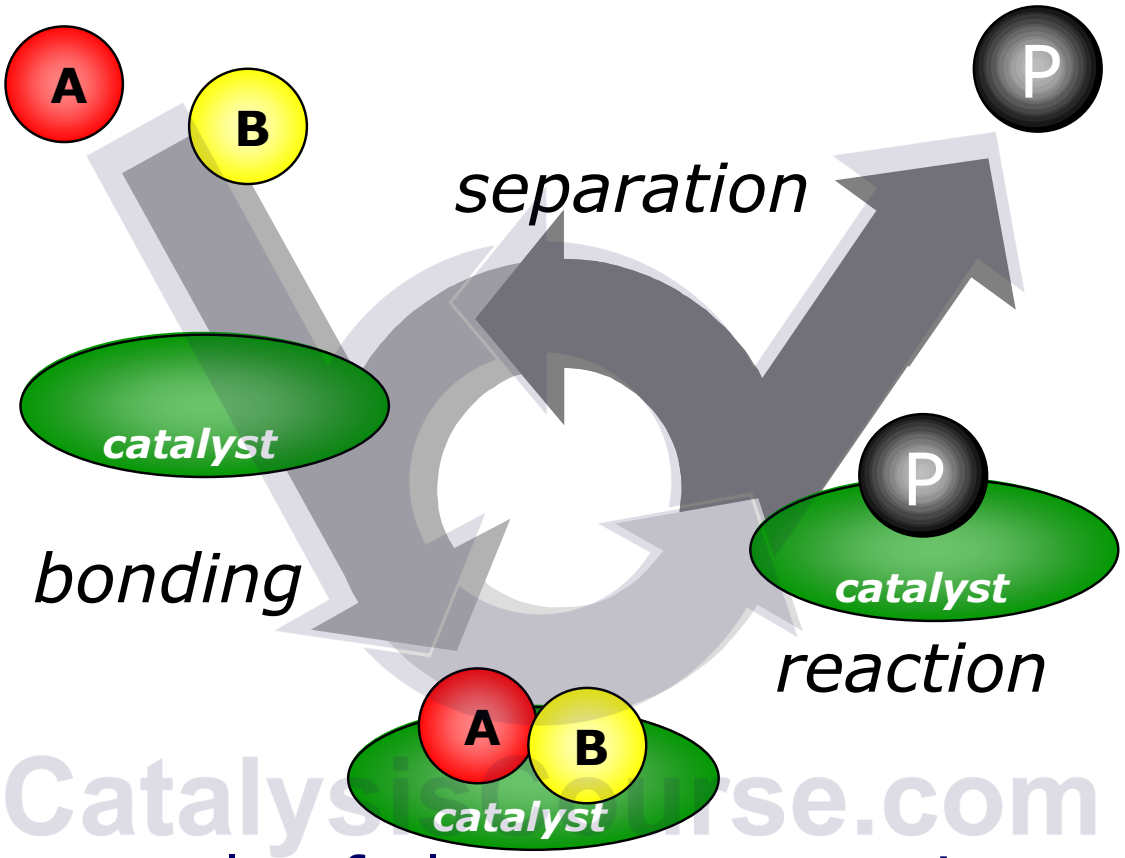
Technische Universiteit Eindhoven University of Technology

Where innovation starts

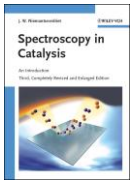


How does a catalytic reaction proceed?

$$A + B \rightarrow P$$



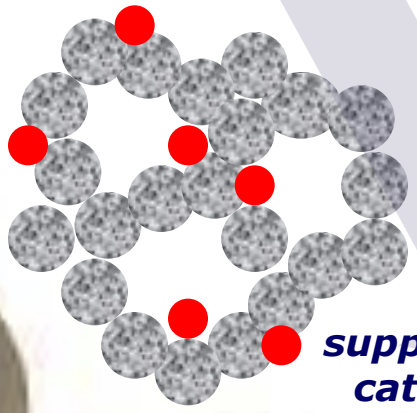
Answer: via a cycle of elementary reaction steps in which molecules react in a complex formed with sites on the catalyst, which are regenerated at the end of the cycle



What is a catalyst?

Catalysts

- increase the rate of a reaction
- without being consumed in the process



supported catalyst

catalyst pellets and extrudates
Courtesy Haldor Topsoe

- ✓ offer alternative, energetically favorable pathways for reactions
- ✓ enable reactions to occur under industrially achievable conditions
- ✓ allow selective production routes without or with less undesirable byproducts
- ✓ are the work horses of the chemical industry
- ✓ are the key enablers for sustainable (green)production

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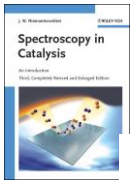


length and time scales in catalytic processes

10 mm

1 nm

shaped catalyst particles

catalytic surface

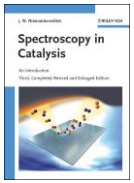
1 m

catalyst bed in a reactor

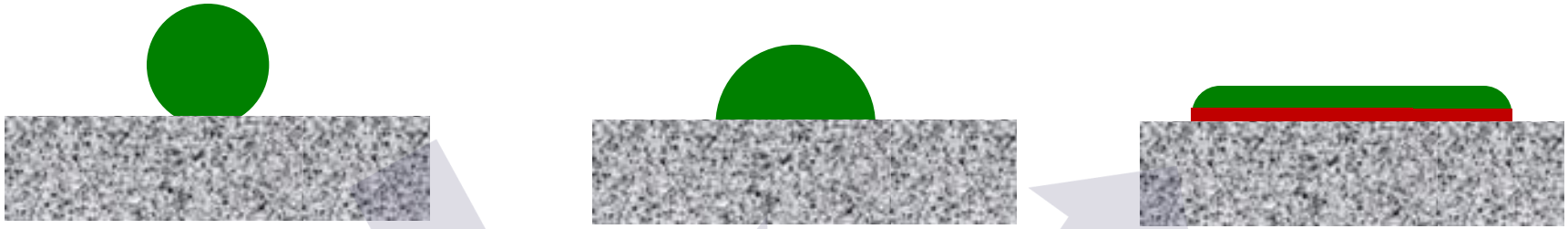
1 μm

catalytically active particles on a support

microscopic ***mesoscopic*** ***macroscopic***

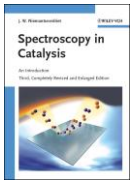


Supported catalyst



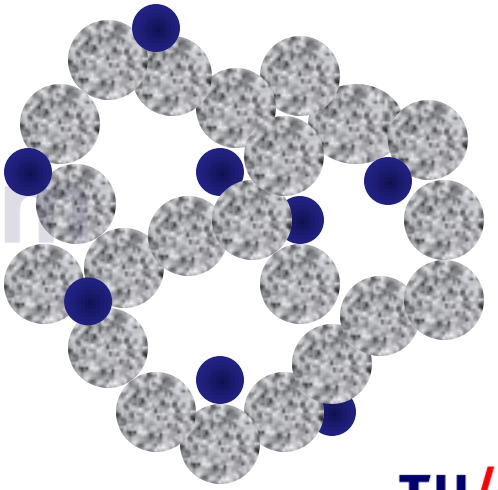
Metal – support combination determines

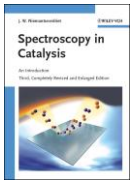
- particle morphology
  - crystal planes exposed
  - steps, kinks, etc
  - type of interface with support
- degree of reduction
- particle size
- stability against sintering
- involvement of support in reactions



Requirements of a successful catalyst

- High activity per unit volume in the reactor
- High selectivity at high conversion; no byproducts
- Long life time
- Regenerable
- Reproducible preparation & activation
- Thermal stability (no sintering)
- High mechanical strength
- High attrition resistance





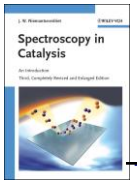
Aims of Catalyst Characterization

Fundamental research:

- composition & structure
- of the catalytic surface
- under reaction conditions
- in atomic detail

Applied research:

- identification of properties that discriminate between poor and successful catalysts



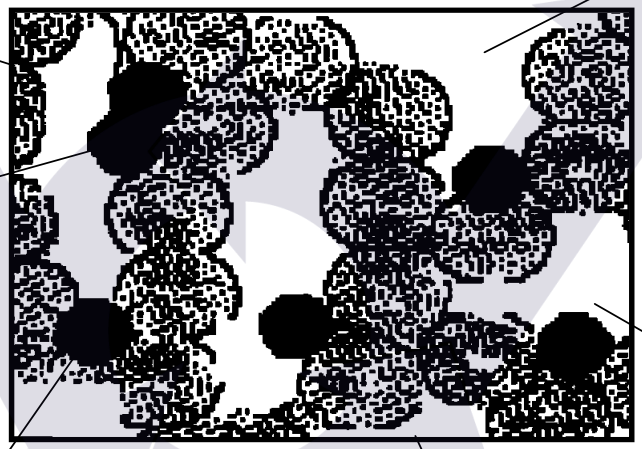
Catalyst Characterization

What do we want to know about a supported catalyst?

Surface Area
Total: BET
Metal: H2 or CO chemisorption
Pore size distribution:
Hg porosimetry

Composition
XPS, XANES, XRD
ICP, AAS

Surface Composition
LEIS, XPS, SIMS



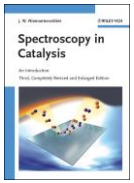
Morphology
Particles: TEM
Overall: SEM

Particle size
Electron Microscopy
H2 chemisorption
XRD line broadening

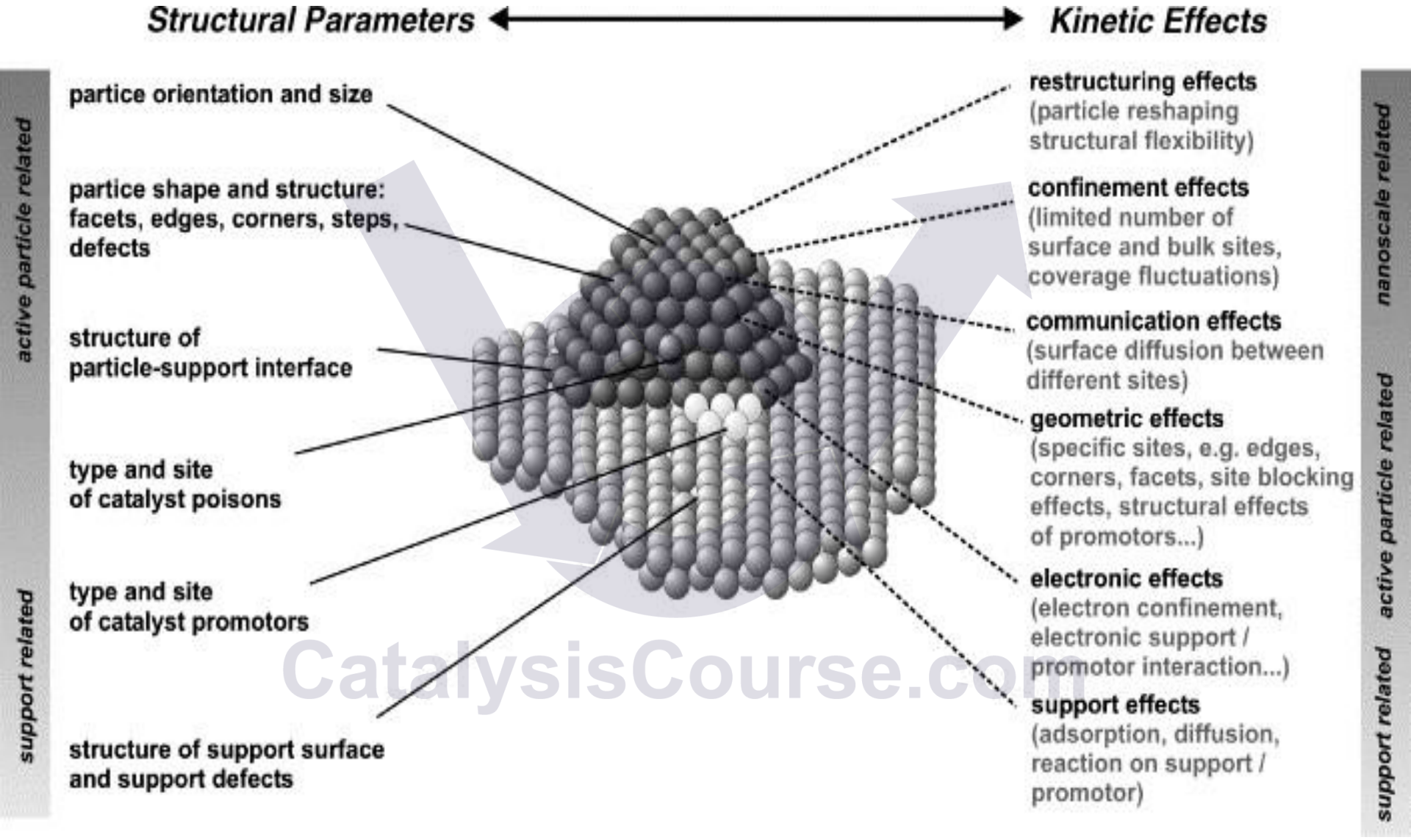
Adsorbed Gases
FTIR, DRIFTS, TPD

Structure
XRD
XPS, EXAFS,TEM

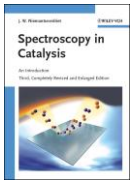
Degree of Reduction
TPR, XPS , XANES



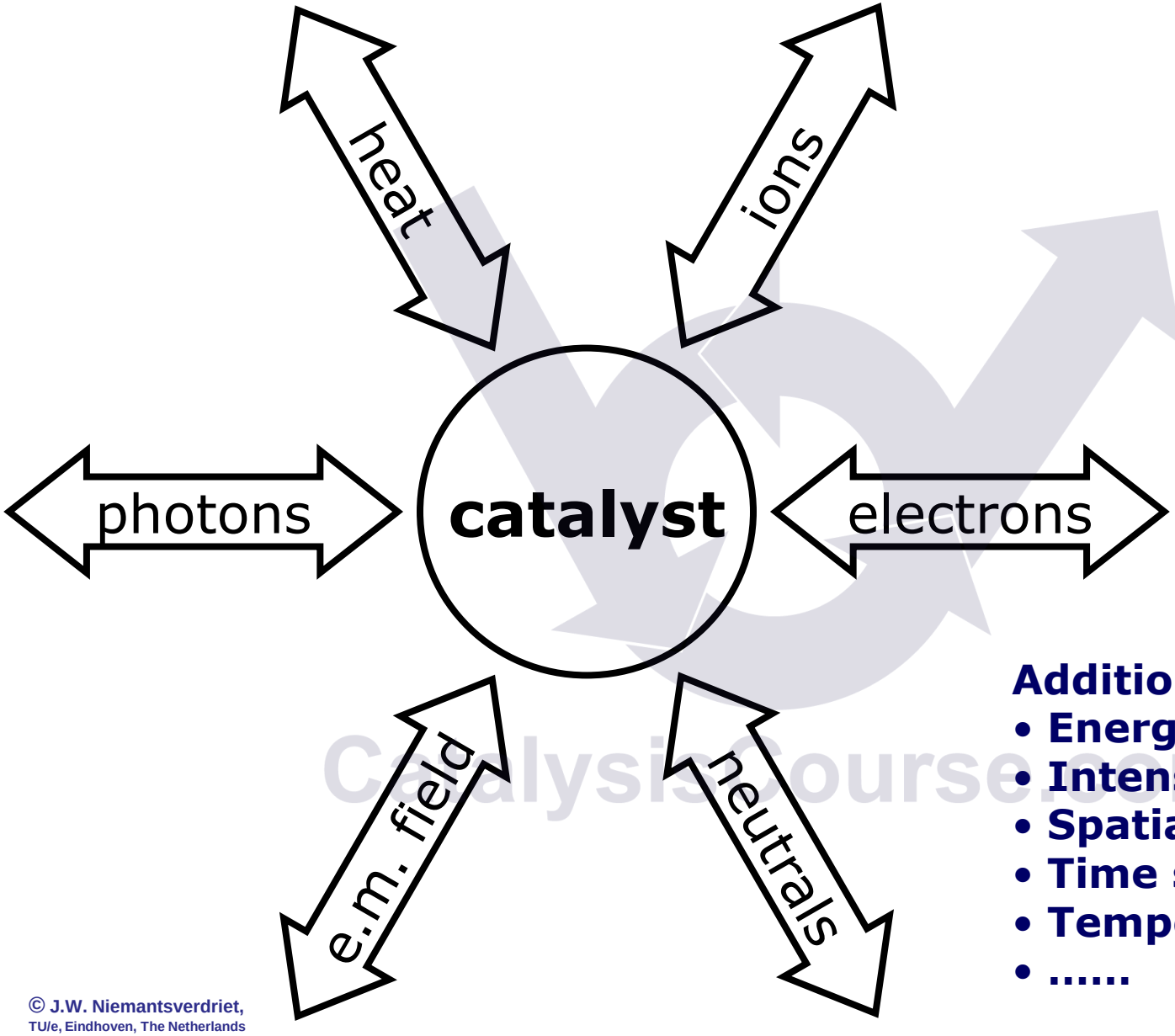
Things that Matter in a Supported Catalyst:



J. Libuda and H.-J. Freund, Surf. Sci. Rep. 57 (2005) 157

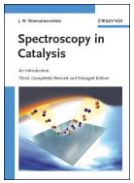


'All' Characterization Techniques can be derived from:



Additional parameters:

- **Energy**
- **Intensity**
- **Spatial configuration**
- **Time structure**
- **Temperature**
- **……**



How often are techniques used

XRD	18.4
Adsorption	17.4
XPS	10.0
TP Techniques	9.2
Infrared	7.3
TEM	6.9
SEM	4.3
UV-vis	4.3
NMR	3.6
Raman	3.2
ESR	2.0
EXAFS	1.9
XANES	1.5
EDX	1.2
Mossbauer	0.8
Calorimetry	0.4
ISS / LEIS	<0.1
Neutron Scattering	<0.1
SIMS	<0.1

Journals:

Applied Catalysis A & B
Catalysis Letters
Journal of Catalysis
Jan 2002 and Oct 2006

Total Number of Articles:

8112

0 5 10 15 20

percentage

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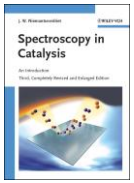

In situ or under vacuum?

	real catalyst	single crystal
reaction conditions	XRD, TP techniques Infrared and Raman EXAFS, XANES, AFM Mossbauer, ESR, NMR	Infrared TP techniques STM,AFM
vacuum	XPS, SIMS, SNMS LEIS, RBS, TEM, SEM	all surface science techniques

Download the handout for this lecture from

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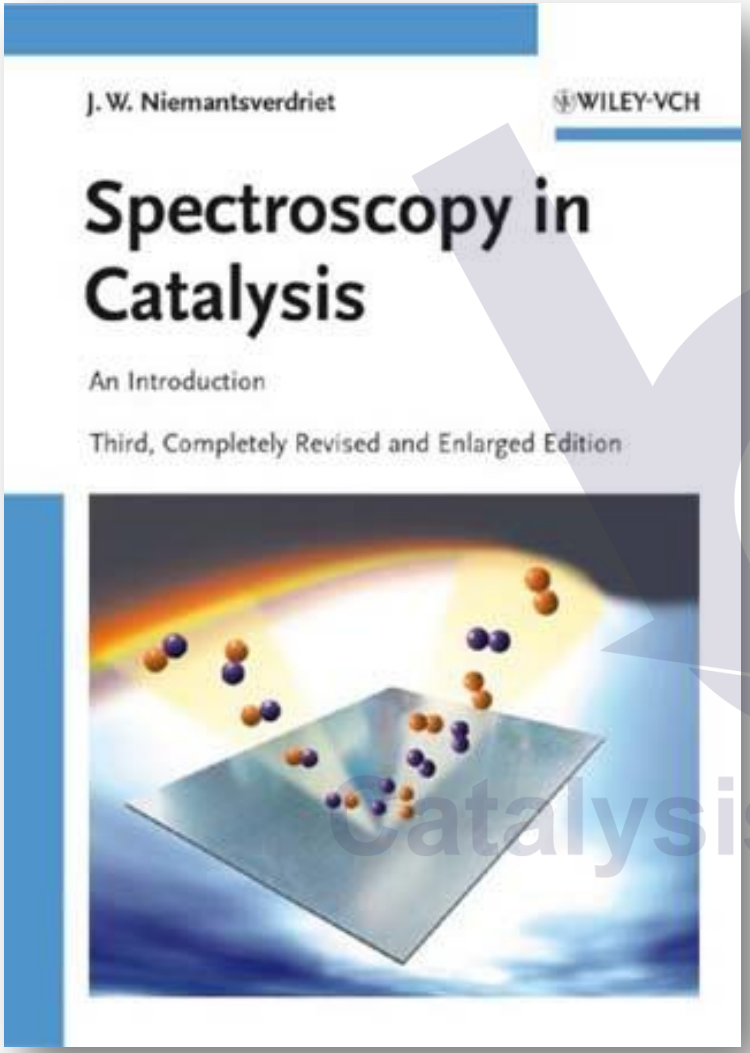


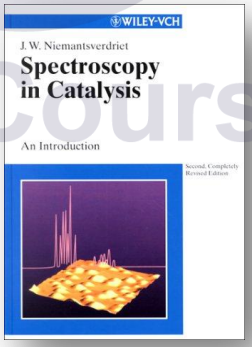
Read more in

Spectroscopy in Catalysis: An Introduction, Third Edition

J. W. Niemantsverdriet

ISBN: 978-3-527-31651-9

NB CHAPTER ONE AVAILABLE
ON WEBSITE COURSE FOR FREE



Version 2000

gives many examples and references to the literature

TU/e Technische Universiteit Eindhoven University of Technology