Sustainability in Organic Chemistry Lab Courses

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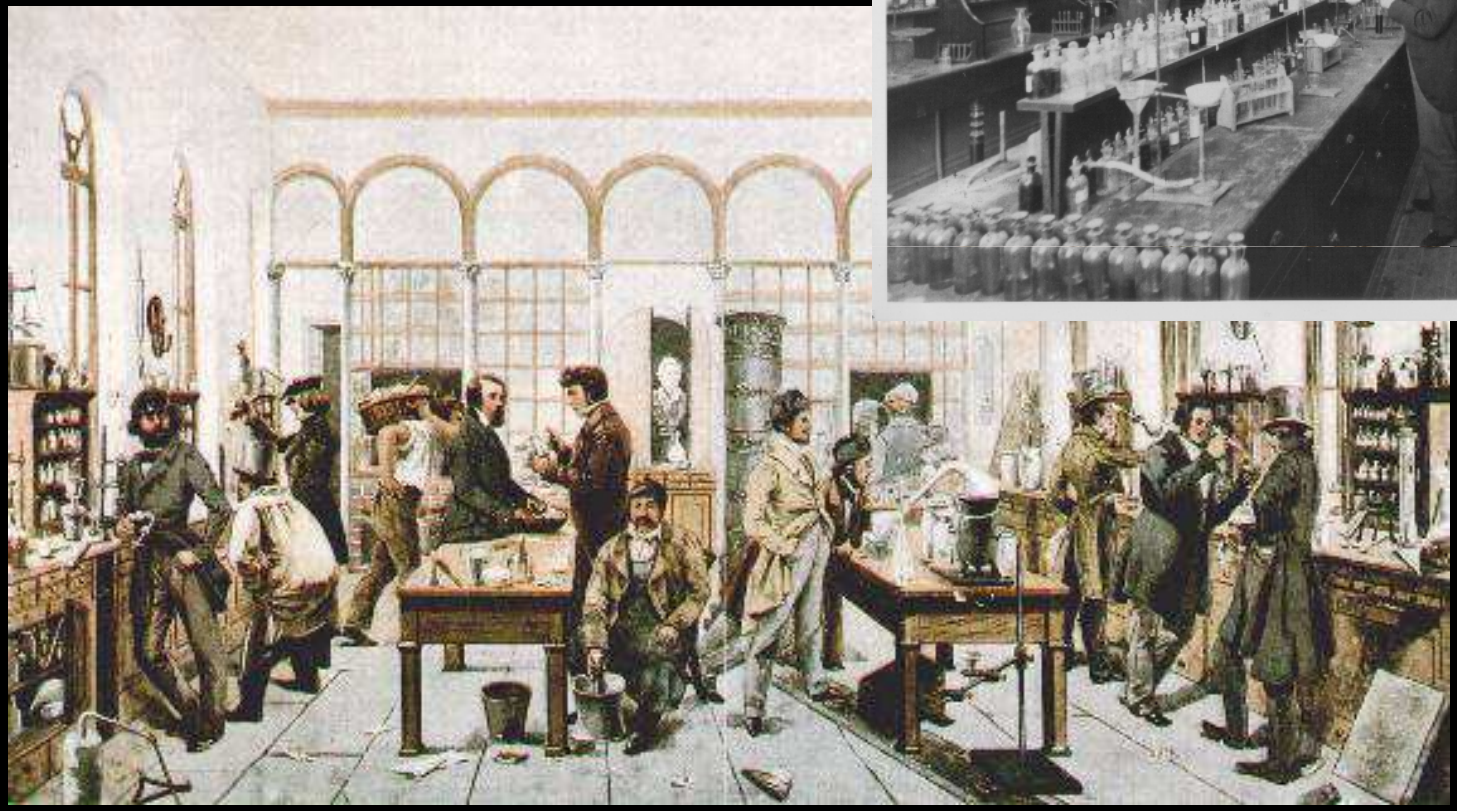
Pharmaceutical Laboratory in 1894

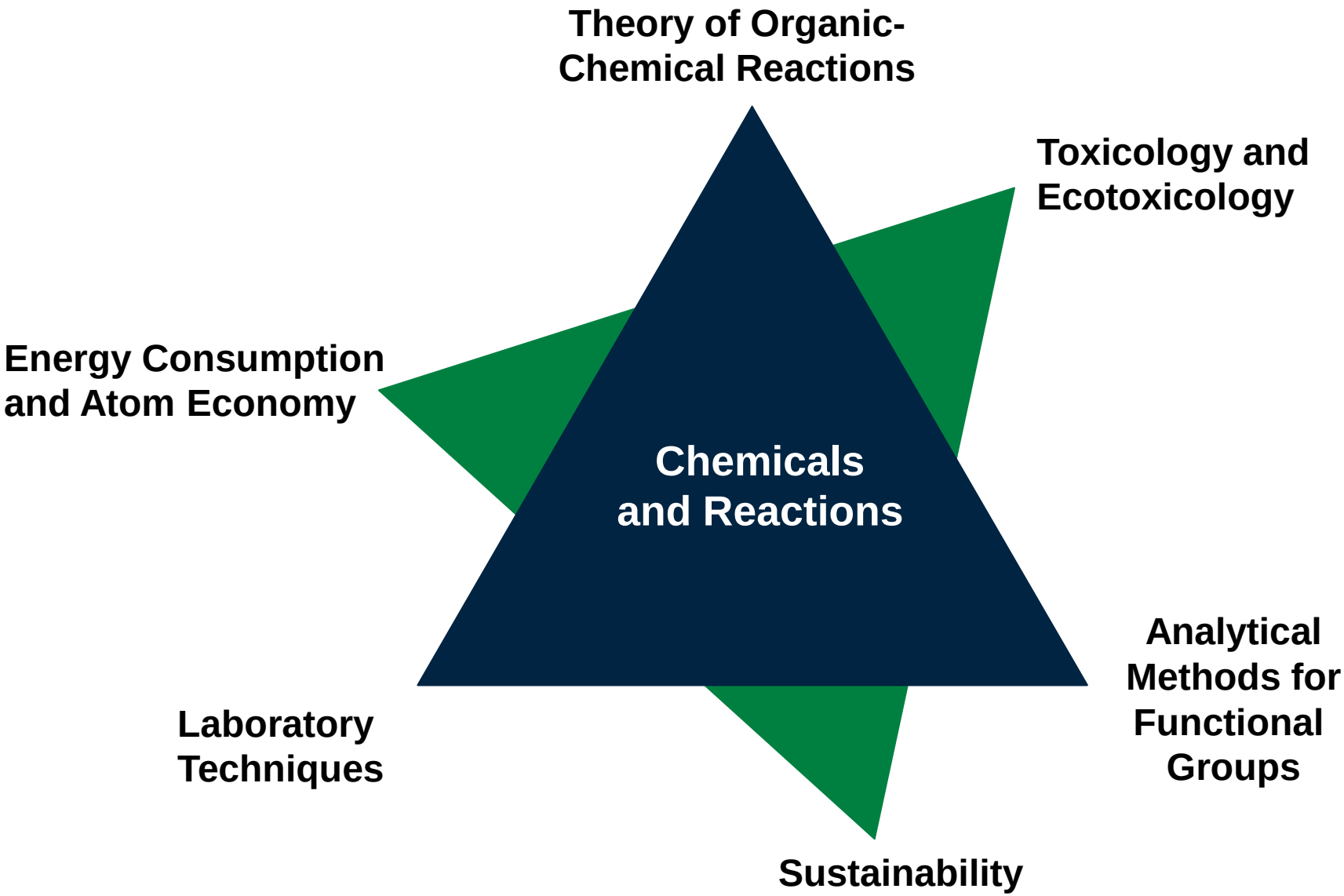


Liebig´s Analytical Laboratory at 1840

Chemical Laboratory
in Vietnam 2004

Chemical Laboratory
in Kazakhstan 2005

Chemical Laboratory
in Indonesia 2006


Theory of Organic-
Chemical Reactions
Toxicology and
Ecotoxicology
Energy Consumption
and Atom Economy
Chemicals
and Reactions
Analytical
Methods for
Functional
Groups
Laboratory
Techniques
Sustainability

New Aspects of Sustainable Chemistry Education

How good does a chemical reaction work?

What is about the atom economy – yields vs. by-products?

State-of-art analyses of raw and final products

Alternative reaction control

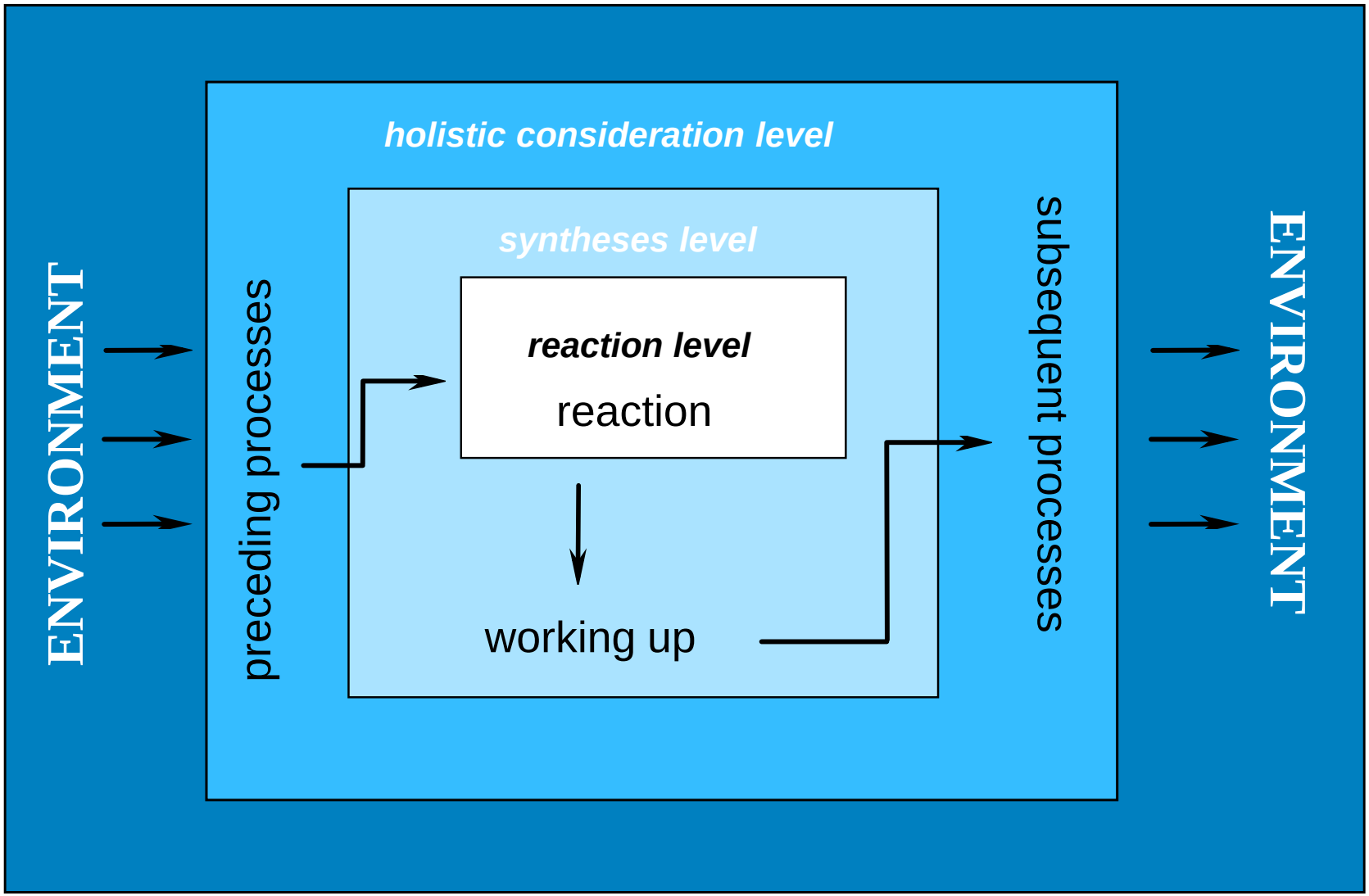
Factors of energy consumption and losses

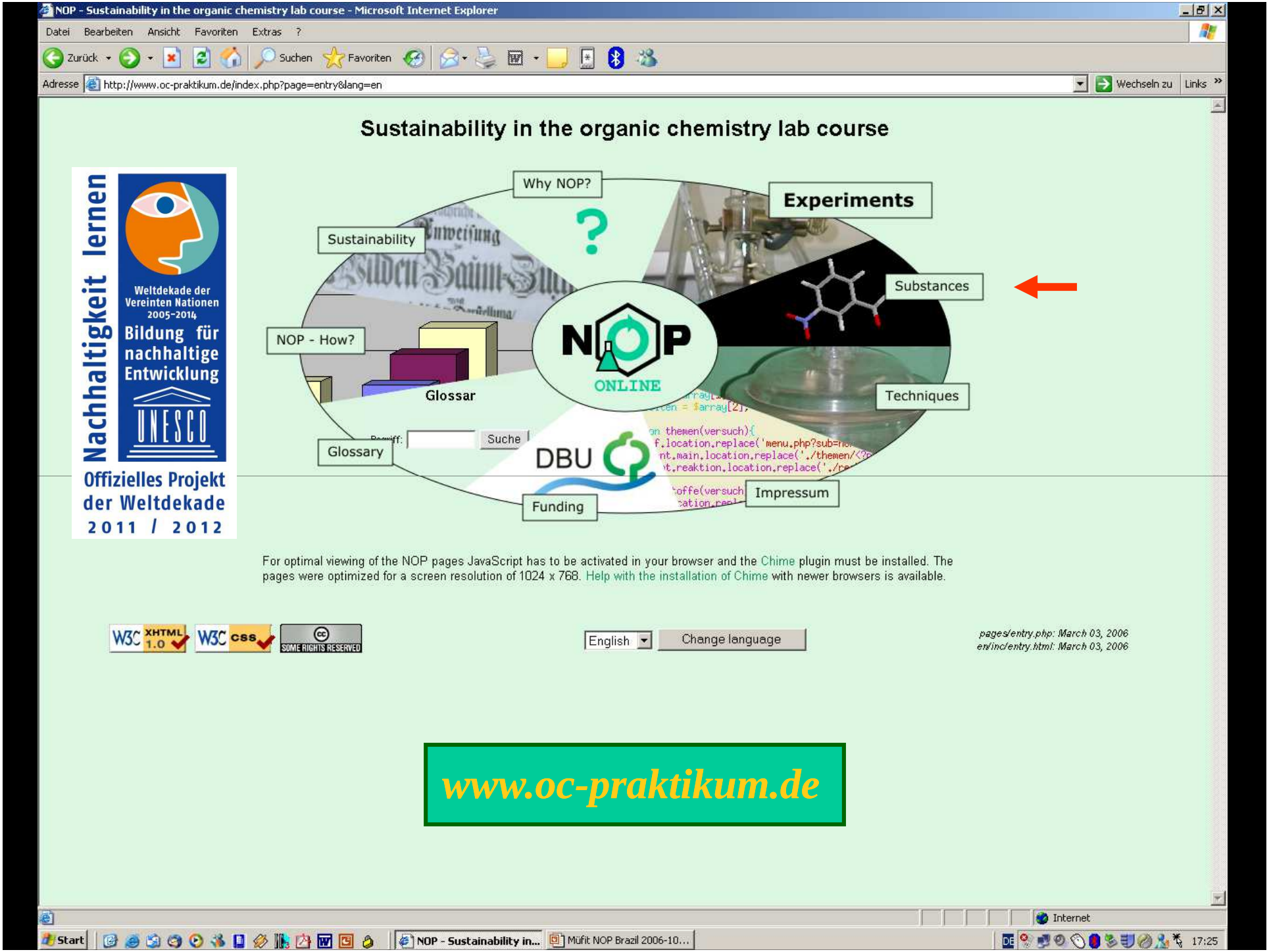
Effect factors for hazard – toxic or not?

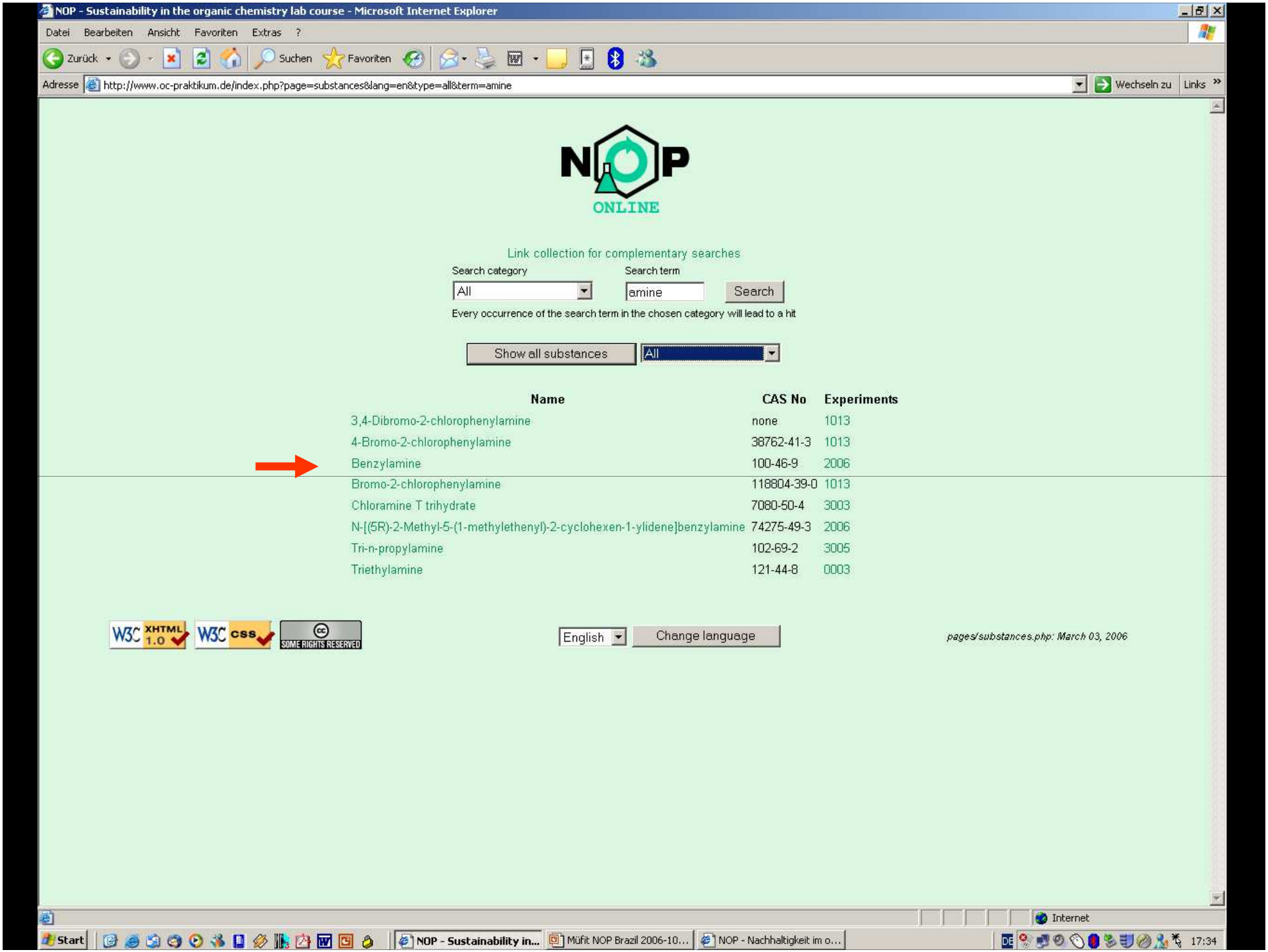
In general:

Which are the factors of environmental acceptability?

Chemicals do not respect national borders !




NOP - Sustainability in the organic chemistry lab course - Microsoft Internet Explorer
Datei Bearbeiten Ansicht Favoriten Extras ?
Zurück Suchen Favoriten
Adresse http://www.oc-praktikum.de/index.php?page=entry&lang=en
Wechseln zu Links
Sustainability in the organic chemistry lab course
Nachhaltigkeit lernen
Weltdekade der Vereinten Nationen 2005-2014
Bildung für nachhaltige Entwicklung
UNESCO
Offizielles Projekt der Weltdekade 2011 / 2012
Why NOP?
Experiments
Sustainability
Substances
NOP - How?
Techniques
Glossar
NOP
ONLINE
Glossary
Suche
DBU
Funding
Impressum
For optimal viewing of the NOP pages JavaScript has to be activated in your browser and the Chime plugin must be installed. The pages were optimized for a screen resolution of 1024 x 768. Help with the installation of Chime with newer browsers is available.
W3C XHTML 1.0
W3C CSS
SOME RIGHTS RESERVED
English
Change language
pages/entry.php: March 03, 2006
en/inc/entry.html: March 03, 2006
www.oc-praktikum.de
Internet
Start
NOP - Sustainability in...
Müfit NOP Brazil 2006-10...
17:25


NOP - Sustainability in the organic chemistry lab course - Microsoft Internet Explorer
Datei Bearbeiten Ansicht Favoriten Extras ?
Zurück Suchen Favoriten
Adresse http://www.oc-praktikum.de/index.php?page=substances&lang=en&type=all&term=amine
Wechseln zu Links
NOP ONLINE
Link collection for complementary searches
Search category
All
Search term
amine
Search
Every occurrence of the search term in the chosen category will lead to a hit
Show all substances
All
Name
CAS No
Experiments
3,4-Dibromo-2-chlorophenylamine none 1013
4-Bromo-2-chlorophenylamine 38762-41-3 1013
Benzylamine 100-46-9 2006
Bromo-2-chlorophenylamine 118804-39-0 1013
Chloramine T trihydrate 7080-50-4 3003
N-[(5R)-2-Methyl-5-(1-methylethenyl)-2-cyclohexen-1-ylidene]benzylamine 74275-49-3 2006
Tri-n-propylamine 102-69-2 3005
Triethylamine 121-44-8 0003
W3C XHTML 1.0
W3C CSS
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English
Change language
pages/substances.php: March 03, 2006
Internet
Start
NOP - Sustainability in...
Müfit NOP Brazil 2006-10...
NOP - Nachhaltigkeit im o...
17:34

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Datei Bearbeiten Ansicht Favoriten Extras ?

Zurück Suchen Favoriten

Adresse http://www.oc-praktikum.de/index.php?page=substance&chent_id=187&view=3D&lang=en

Wechseln zu Links

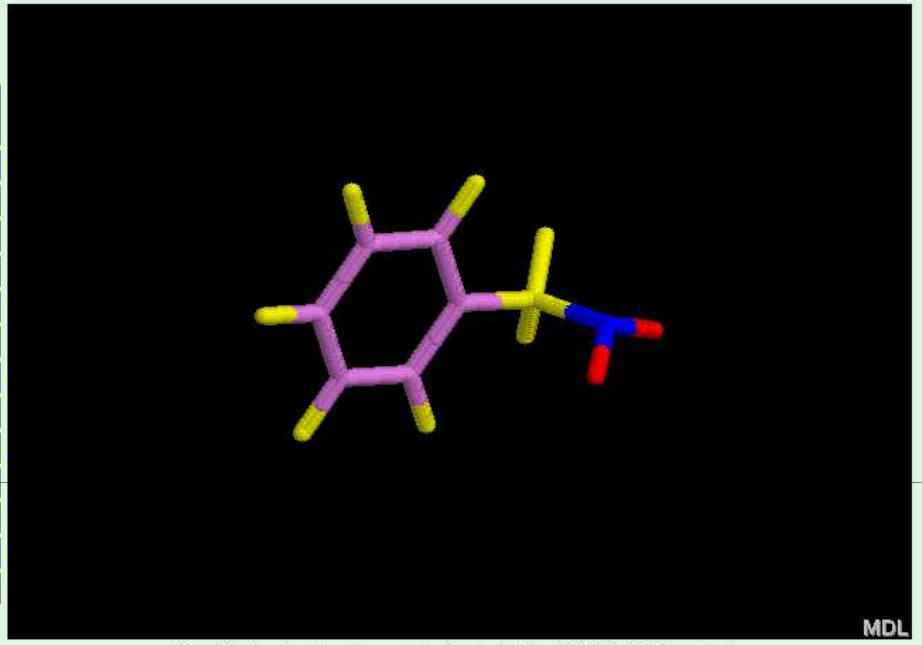
NOP ONLINE

Identity
3D Structure
Safety classifications

Benzylamine [100-46-9]

Go | Legend:

- Topology
- Volume
- Dot surface
- Surface
- Chirality
- MIP - no CT
- MIP - incl. CT
- Chameleon
- Charges
- Potential
- Lipophilicity
- CPK-Colours
- Transparent



The 3D structure has been optimized with the MOPAC PM3 method.

W3C CSS

English Change language

pages/substance.php: March 03, 2006
views/3D.php: March 03, 2006

Chime script completed.

Internet

Start NOP - Sustainability in... Müfit NOP Brazil 2006-10... NOP - Nachhaltigkeit im o... 17:42

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Datei Bearbeiten Ansicht Favoriten Extras ?

Zurück Suchen Favoriten

Adresse http://www.oc-praktikum.de/index.php?page=substance&chent_id=187&view=3D&lang=en

Wechseln zu Links

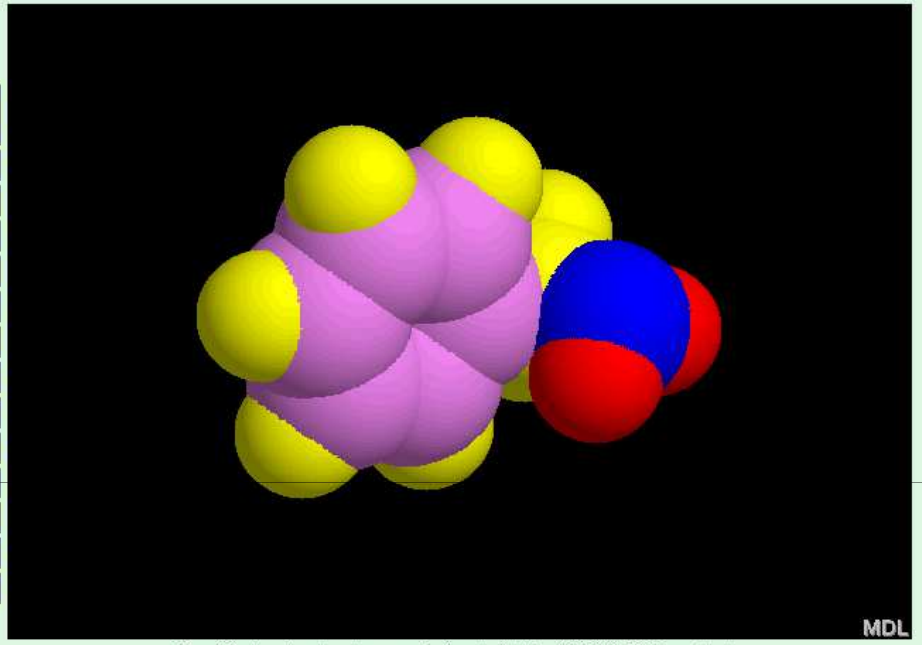
NOP ONLINE

Identity
3D Structure
Safety classifications

Benzylamine [100-46-9]

Go | Legend:

- Topology
- Volume
- Dot surface
- Surface
- Chirality
- MIP - no CT
- MIP - incl. CT
- Chameleon
- Charges
- Potential
- Lipophilicity
- CPK-Colours
- Transparent



The 3D structure has been optimized with the MOPAC PM3 method.

W3C CSS SOME RIGHTS RESERVED

English Change language

pages/substance.php: March 03, 2006
views/3D.php: March 03, 2006

Chime script completed.

Internet

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Zurück Suchen Favoriten

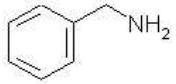
Adresse http://www.oc-praktikum.de/index.php?page=substance&chent_id=187&view=classifications&lang=en

Wechseln zu Links

NOP ONLINE

Identity
3D Structure
Safety classifications

Benzylamine [100-46-9]



	value	comment	source
Permissible air concentration:	not assigned		
Effect factor after TRGS 440 (German)	100		TRGS 440 (German), 2001
Water pollution class:	2		Catalog of water polluting substances (German)
Hazard symbols:	C		EU
Risk phrases:	R 21/22-34		EU
Safety phrases:	S 1/2-26-36/37/39-45		EU
Data availability	Toxicity and Ecotoxicity data		AG Jastorff

http://www.oc-praktikum.de - NOP - Glossary - Microsoft Internet Explorer

effect factor

The effect factor is a dimensionless number that is determined according to a method outlined in the German Technical Rules for Hazardous Substances (TRGS) 440. This method uses as input the known R phrases and the German threshold limit values. It also provides a classification of substances with not fully determined or unknown dangerous properties.

[HTML] **Evaluation of chemical substances**

English Change language

Fertig Internet

English Change language

pages/substance.php: March 03, 2006
views/classifications.php: March 03, 2006

Internet

Start NOP - Sustainability in t... Müfit NOP Brazil 2006-1... NOP - Nachhaltigkeit im ... http://www.oc-prakt... 17:48

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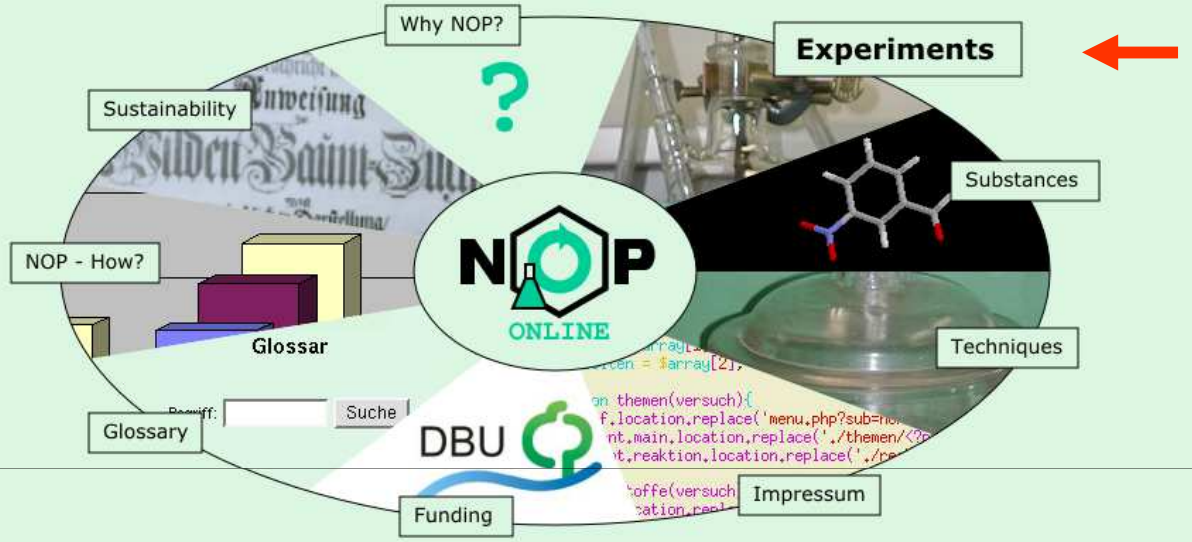
Datei Bearbeiten Ansicht Favoriten Extras ?

Zurück Suchen Favoriten

Adresse http://www.oc-praktikum.de/index.php?page=entry&lang=en Wechseln zu Links

Sustainability in the organic chemistry lab course



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English Change language

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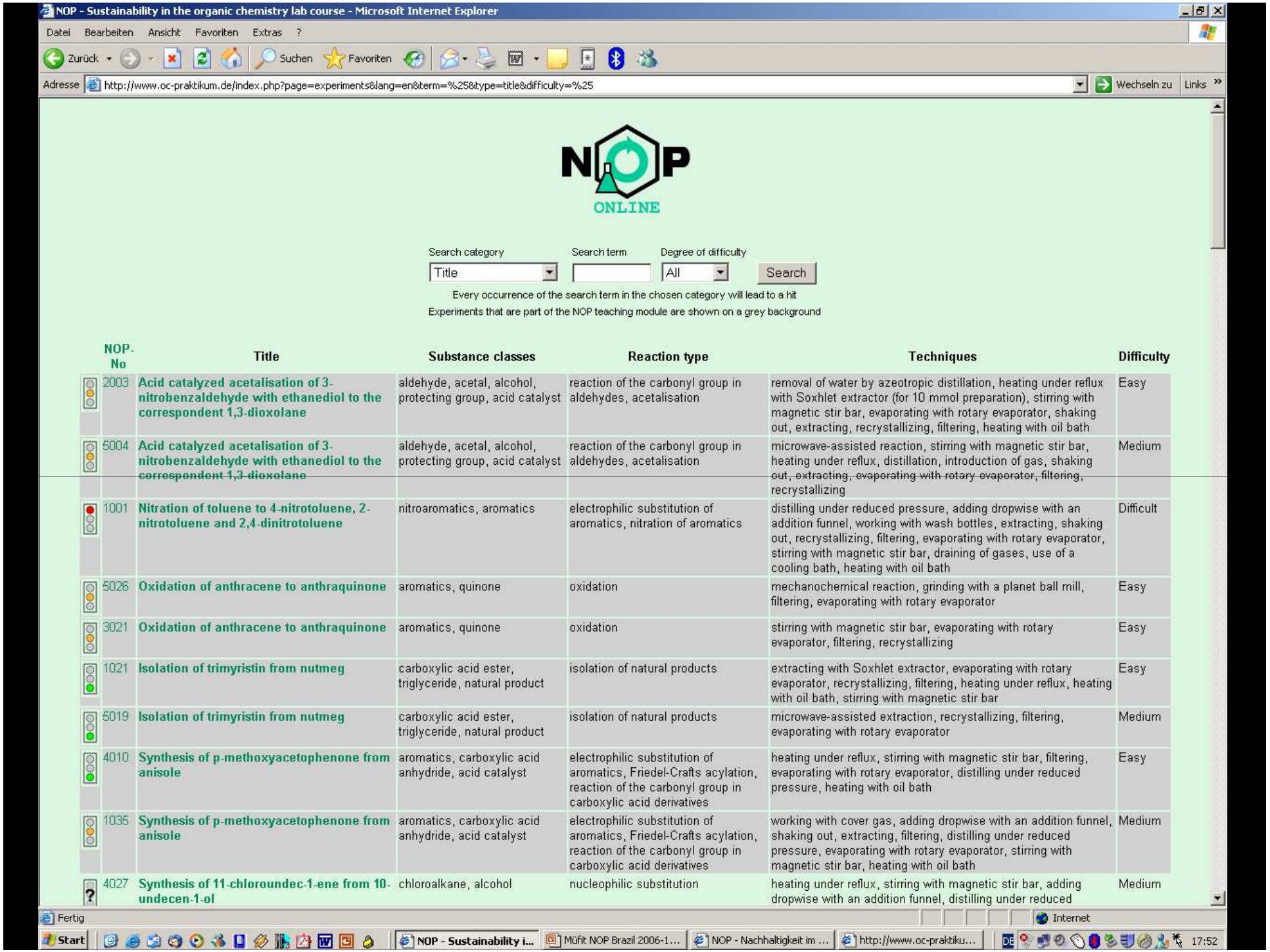
Internet

Start NOP - Sustainability in... Müfit NOP Brazil 2006-10... 17:25


NOP - Sustainability in the organic chemistry lab course - Microsoft Internet Explorer
Datei Bearbeiten Ansicht Favoriten Extras ?
Zurück Suchen Favoriten
Adresse http://www.oc-praktikum.de/index.php?page=experiments&lang=en&term=%25&type=title&difficulty=%25
Wechseln zu Links
NOP ONLINE
Search category
Title
Search term
Degree of difficulty
All
Search
Every occurrence of the search term in the chosen category will lead to a hit
Experiments that are part of the NOP teaching module are shown on a grey background
NOP-No
Title
Substance classes
Reaction type
Techniques
Difficulty
2003 Acid catalyzed acetalisation of 3-nitrobenzaldehyde with ethanediol to the correspondent 1,3-dioxolane
aldehyde, acetal, alcohol, protecting group, acid catalyst
reaction of the carbonyl group in aldehydes, acetalisation
removal of water by azeotropic distillation, heating under reflux with Soxhlet extractor (for 10 mmol preparation), stirring with magnetic stir bar, evaporating with rotary evaporator, shaking out, extracting, recrystallizing, filtering, heating with oil bath
Easy
5004 Acid catalyzed acetalisation of 3-nitrobenzaldehyde with ethanediol to the correspondent 1,3-dioxolane
aldehyde, acetal, alcohol, protecting group, acid catalyst
reaction of the carbonyl group in aldehydes, acetalisation
microwave-assisted reaction, stirring with magnetic stir bar, heating under reflux, distillation, introduction of gas, shaking out, extracting, evaporating with rotary evaporator, filtering, recrystallizing
Medium
1001 Nitration of toluene to 4-nitrotoluene, 2-nitrotoluene and 2,4-dinitrotoluene
nitroaromatics, aromatics
electrophilic substitution of aromatics, nitration of aromatics
distilling under reduced pressure, adding dropwise with an addition funnel, working with wash bottles, extracting, shaking out, recrystallizing, filtering, evaporating with rotary evaporator, stirring with magnetic stir bar, draining of gases, use of a cooling bath, heating with oil bath
Difficult
5026 Oxidation of anthracene to anthraquinone
aromatics, quinone
oxidation
mechanochemical reaction, grinding with a planet ball mill, filtering, evaporating with rotary evaporator
Easy
3021 Oxidation of anthracene to anthraquinone
aromatics, quinone
oxidation
stirring with magnetic stir bar, evaporating with rotary evaporator, filtering, recrystallizing
Easy
1021 Isolation of trimyristin from nutmeg
carboxylic acid ester, triglyceride, natural product
isolation of natural products
extracting with Soxhlet extractor, evaporating with rotary evaporator, recrystallizing, filtering, heating under reflux, heating with oil bath, stirring with magnetic stir bar
Easy
5019 Isolation of trimyristin from nutmeg
carboxylic acid ester, triglyceride, natural product
isolation of natural products
microwave-assisted extraction, recrystallizing, filtering, evaporating with rotary evaporator
Medium
4010 Synthesis of p-methoxyacetophenone from anisole
aromatics, carboxylic acid anhydride, acid catalyst
electrophilic substitution of aromatics, Friedel-Crafts acylation, reaction of the carbonyl group in carboxylic acid derivatives
heating under reflux, stirring with magnetic stir bar, filtering, evaporating with rotary evaporator, distilling under reduced pressure, heating with oil bath
Easy
1035 Synthesis of p-methoxyacetophenone from anisole
aromatics, carboxylic acid anhydride, acid catalyst
electrophilic substitution of aromatics, Friedel-Crafts acylation, reaction of the carbonyl group in carboxylic acid derivatives
working with cover gas, adding dropwise with an addition funnel, shaking out, extracting, filtering, distilling under reduced pressure, evaporating with rotary evaporator, stirring with magnetic stir bar, heating with oil bath
Medium
4027 Synthesis of 11-chloroundec-1-ene from 10-undecen-1-ol
chloroalkane, alcohol
nucleophilic substitution
heating under reflux, stirring with magnetic stir bar, adding dropwise with an addition funnel, distilling under reduced
Fertig
Internet
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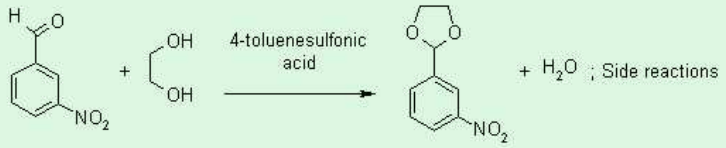
http://www.oc-praktiku...
17:52

NOP ONLINE

NOP-Nr: 2003
Alternative: 5004



Acid catalyzed acetalisation of 3-nitrobenzaldehyde with ethanediol to the correspondent 1,3-dioxolane

Synthesis instructions as PDF file for printing

Batch scale: (•) 0.1 mol ( ) 0.01 mol 3-Nitrobenzaldehyde

Reaction

3-Nitrobenzaldehyde (15.1 g, 100 mmol), ethanediole (6.83 g, 6.20 mL, 110 mmol) and 4-toluenesulfonic acid monohydrate (1.00 g, 5.30 mmol) are dissolved in cyclohexane (200 mL) in a dry 500 mL round bottom flask equipped with magnetic stirring bar, Dean Stark trap and reflux condenser. The reaction mixture is refluxed until no more water is collected in the Dean-Stark trap (approx. 2-3 h).

Work up

The hot reaction mixture is poured into another 500 mL round bottom flask to separate it from an oily sediment (800 mg) which has formed at the bottom of the reaction vessel. The sediment consists predominantly of product, starting material and 4-toluenesulfonic acid (^{1}H-NMR spectrum). The solvent of the decanted solution is directly removed with a rotary evaporator . A yellow crystalline solid remains as crude product.

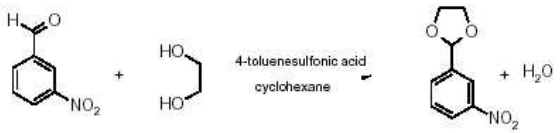
Crude product yield: 19.7 g; melting point 50-52 °C; Purity according to GC: 95% acetale + 4% aldehyde

In order to remove unreacted aldehyde as hydrogensulfite adduct, the crude product is dissolved in 200 mL *tert*-butyl methyl ether and extracted once with 20 mL saturated aqueous sodium hydrogen sulfite solution. The organic phase is dried over sodium sulfate, the sodium sulfate is removed by filtration and the solvent is evaporated with a rotary evaporator to yield a nearly colorless crystalline residue.

Yield: 17.9 g; melting point 57-58 °C; Purity according to GC: more than 99%.

The product is for most uses pure enough. If further purification is required, recrystallization from a solvent mixture of cyclohexane / *tert*-butyl methyl ether in a ratio of 1:1 (approximately 45 mL) can be carried out. The solution should be allowed to stand covered at room temperature until crystals form (if needed 1 to 2 days). If the solution is cooled quickly in an ice bath, only an oil generally forms. After cooling for a short time in an ice bath, the crystals are filtered and dried until constant mass is achieved in a desiccator at reduced pressure.

2003 Acid catalyzed acetalisation of 3-nitrobenzaldehyde with ethanediol to the correspondent 1,3-dioxolane



$C_7H_5NO_3$ (151.1) | $C_2H_6O_2$ (62.1) | $C_7H_8O_3S \cdot H_2O$ (190.2) | $C_9H_9NO_4$ (195.2)

Classification

Reaction types and substance classes
reaction of the carbonyl group in aldehydes, acetalisation
aldehyde, acetal, alcohol, protecting group, acid catalyst

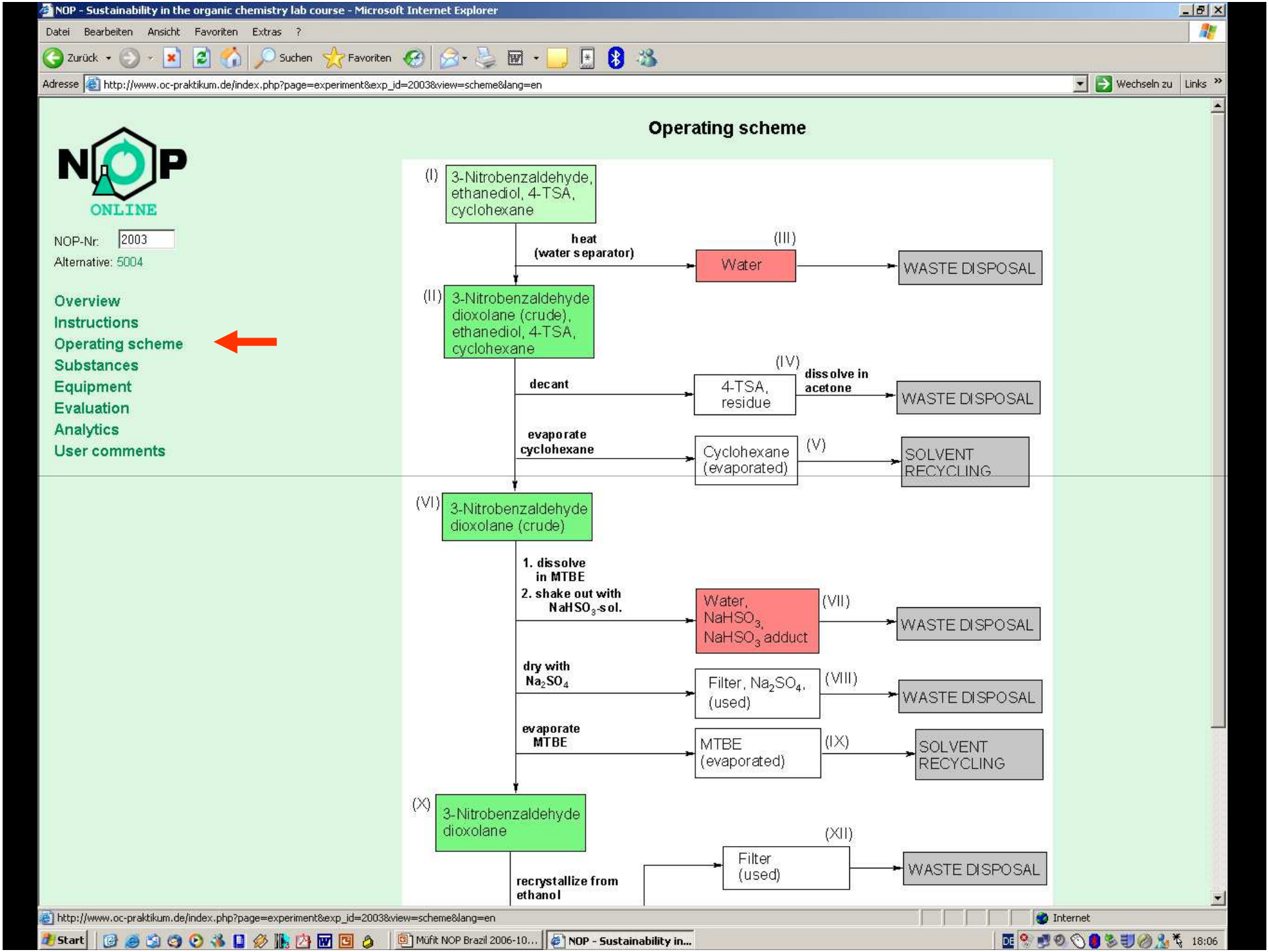
Work methods
removal of water by azeotropic distillation, heating under reflux with Soxhlet extractor (for 10 mmol batch scale), stirring with magnetic stir bar, evaporating with rotary evaporator, shaking out, extracting, recrystallizing, filtering, heating with oil bath

Instruction (batch scale 100 mmol)

Equipment
500 mL round-bottom flask, water separator, reflux condenser, heatable magnetic stirrer with magnetic stir bar, separating funnel, rotary evaporator, suction flask, suction filter, desiccator, oil bath

Substances

3-nitrobenzaldehyde (mp 58 °C; product from NOP-Nr. 1003)	15.1 g (100 mmol)
ethanediol (bp 198 °C)	6.83 g (6.20 mL, 110 mmol)
4-toluenesulfonic acid monohydrate (mp 103-105 °C)	190 mg (1.00 mmol)
cyclohexane (bp 81 °C)	200 mL
tert-butyl methyl ether (bp 55 °C)	200 mL
sodium disulfite	about 13 g (for 20 mL saturated aqueous $NaHSO_3$-solution)
sodium sulfate for drying	about 5 g
cyclohexane (bp 81 °C) for recrystallization	about 30 mL
tert-butyl methyl ether (bp 55 °C) for recrystallization	about 30 mL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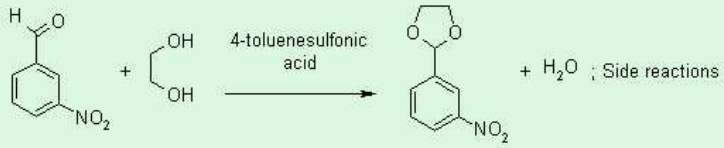
Datei Bearbeiten Ansicht Favoriten Extras ?
Zurück Suchen Favoriten
Adresse http://www.oc-praktikum.de/index.php?page=experiment&exp_id=2003&view=scheme&lang=en
Wechseln zu Links
NOP ONLINE
NOP-Nr: 2003
Alternative: 5004
Overview
Instructions
Operating scheme
Substances
Equipment
Evaluation
Analytics
User comments
Operating scheme
(I) 3-Nitrobenzaldehyde, ethanediol, 4-TSA, cyclohexane
heat (water separator)
(III) Water
WASTE DISPOSAL
(II) 3-Nitrobenzaldehyde dioxolane (crude), ethanediol, 4-TSA, cyclohexane
decant
(IV) 4-TSA, residue
dissolve in acetone
WASTE DISPOSAL
evaporate cyclohexane
Cyclohexane (evaporated)
(V)
SOLVENT RECYCLING
(VI) 3-Nitrobenzaldehyde dioxolane (crude)
1. dissolve in MTBE
2. shake out with NaHSO3-sol.
Water, NaHSO3, NaHSO3 adduct
(VII)
WASTE DISPOSAL
dry with Na2SO4
Filter, Na2SO4, (used)
(VIII)
WASTE DISPOSAL
evaporate MTBE
MTBE (evaporated)
(IX)
SOLVENT RECYCLING
(X) 3-Nitrobenzaldehyde dioxolane
(XII)
Filter (used)
WASTE DISPOSAL
recrystallize from ethanol
http://www.oc-praktikum.de/index.php?page=experiment&exp_id=2003&view=scheme&lang=en
Internet
Start
Müfit NOP Brazil 2006-10...
NOP - Sustainability in...
18:06

NOP ONLINE

NOP-Nr: 2003

Alternative: 5004



Substances required

Batch scale: (•) 0.1 mol ( ) 0.01 mol 3-Nitrobenzaldehyde

Educts		Amount	Risk	Safety
3-Nitrobenzaldehyde	Xn	15.1 g	R 22-36/37/38	S 22-24/25-26-36
1,2-Ethanediol	Xn	6.83 g	R 22	S 2
Catalyst		**Amount**	**Risk**	**Safety**
4-Toluenesulfonic acid monohydrate	Xi	0.19 g	R 36/37/38	S 2-26-37
Solvents		**Amount**	**Risk**	**Safety**
Cyclohexane	F Xn N	~ 230 mL	R 11-38-50/53-65-67	S 2-9-16-33-60-61-62
tert-Butyl methyl ether	F Xi	230 mL	R 11-38	S 2-9-16-24
Others		**Amount**	**Risk**	**Safety**
Sodium disulfite	Xn	~ 13 g	R 22-31-41	S 2-26-39-46
Sodium sulfate	Xi	~ 5 g	R 36/37/38	S 26-36
Molecular sieve 4A	Xi	0 g	R 36/37/38	S 24/25
Solvents for analysis		**Amount**	**Risk**	**Safety**
tert-Butyl methyl ether	F Xi	5 mL	R 11-38	S 2-9-16-24

pages/experiment.php: March 03, 2006
views/required.php: March 03, 2006

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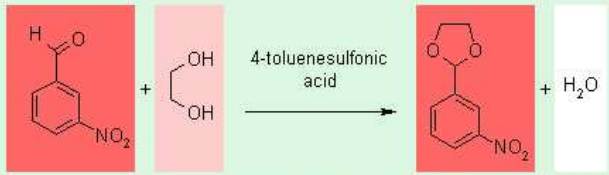
NOP-Nr: 2003
Alternative: 5004



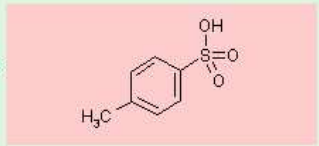
; Side reactions

Effect factors TRGS 440

Effect factor 0 | Effect factor >0 to 10 | Effect factor >10 to 100 | Effect factor >100 to 1000 | Effect factor >1000 to 50000

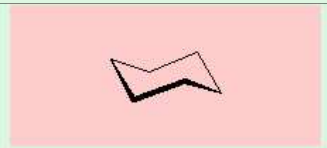
Catalyst

4-Toluenesulfonic acid monohydrate
Effect factor: 5

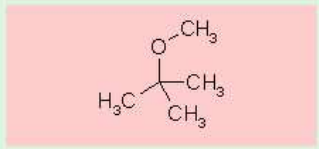


Solvents

Cyclohexane
Effect factor: 5

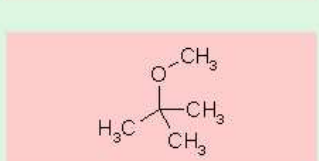


tert-Butyl methyl ether
Effect factor: 5



Solvents for analysis

tert-Butyl methyl ether
Effect factor: 5



Others

Sodium disulfite
Effect factor: 100

$NaHSO_3$

Sodium sulfate
Effect factor: 5

Na_2SO_4

Molecular sieve 4A
Effect factor: 5

$Na_xAl_ySi_zO_n$

English Change language

pages/experiment.php: March 03, 2006
views/effect_factors.php: March 03, 2006

NOP - Sustainability in the organic chemistry lab course - Microsoft Internet Explorer

Datei Bearbeiten Ansicht Favoriten Extras ?

Zurück Suchen Favoriten

Adresse http://www.oc-praktikum.de/index.php?page=experiment&exp_id=2003&view=equipment&lang=en

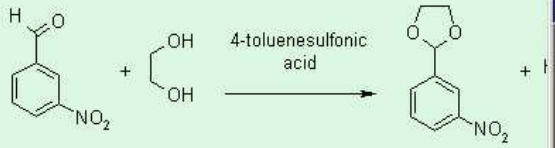
Wechseln zu Links

NOP ONLINE

NOP-Nr: 2003

Alternative: 5004

- Overview
- Instructions
- Operating scheme
- Substances
- Equipment
- Evaluation
- Analytics
- User comments



Equipment

Batch scale: 0.1 mol 0.01 mol 3-Nitrobenzalc


round bottom flask 500 mL


ater separator


reflux condenser


heatable magnetic


separating funnel


rotary evaporator


suction flask


suction filter


exsiccator with drying agent


oil bath

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English Change language

http://www.oc-praktikum.de - water separator - Micr...

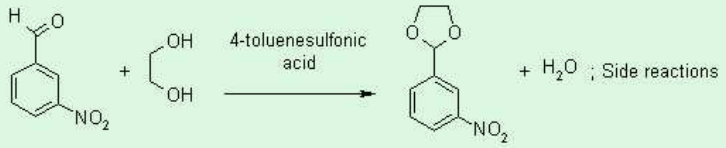
Fertig Internet

Internet

Start Müfit NOP Brazil 2006-1... NOP - Sustainability in t... Normal : Mehrseitendruck http://www.oc-prakt... 18:15

NOP ONLINE

NOP-Nr: 2003
Alternative: 5004



Evaluation text

The classical variant of the "Acid catalyzed acetalisation of 3-nitrobenzaldehyde with ethanediol to the correspondent 1,3-dioxolane" is an easily performed experiment. The desired product is obtained in high yield and high selectivity. Also the purity of the end product is very high.

The mass efficiency is high to medium, compared to the other NOP experiments and thus evaluates as good. The energy efficiency of the classical experiment is highly dependent on the method of heating.

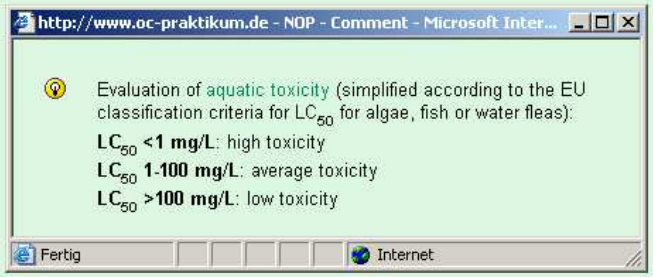
(Eco)toxicological data for the educt 3-nitrobenzaldehyde are incomplete, toxicological data for the product 2-(3-nitrophenyl)-1,3-dioxolane have not been determined at all. According to theoretical prediction methods both product and educt are suspected to have mutagenic, carcinogenic and sensitizing properties. The organic solvents used in this experiment ethanol, cyclohexane and tert-butyl methyl ether exhibit relatively low acute toxicity. Also the inorganic auxiliary materials do not pose significant dangers to human health.

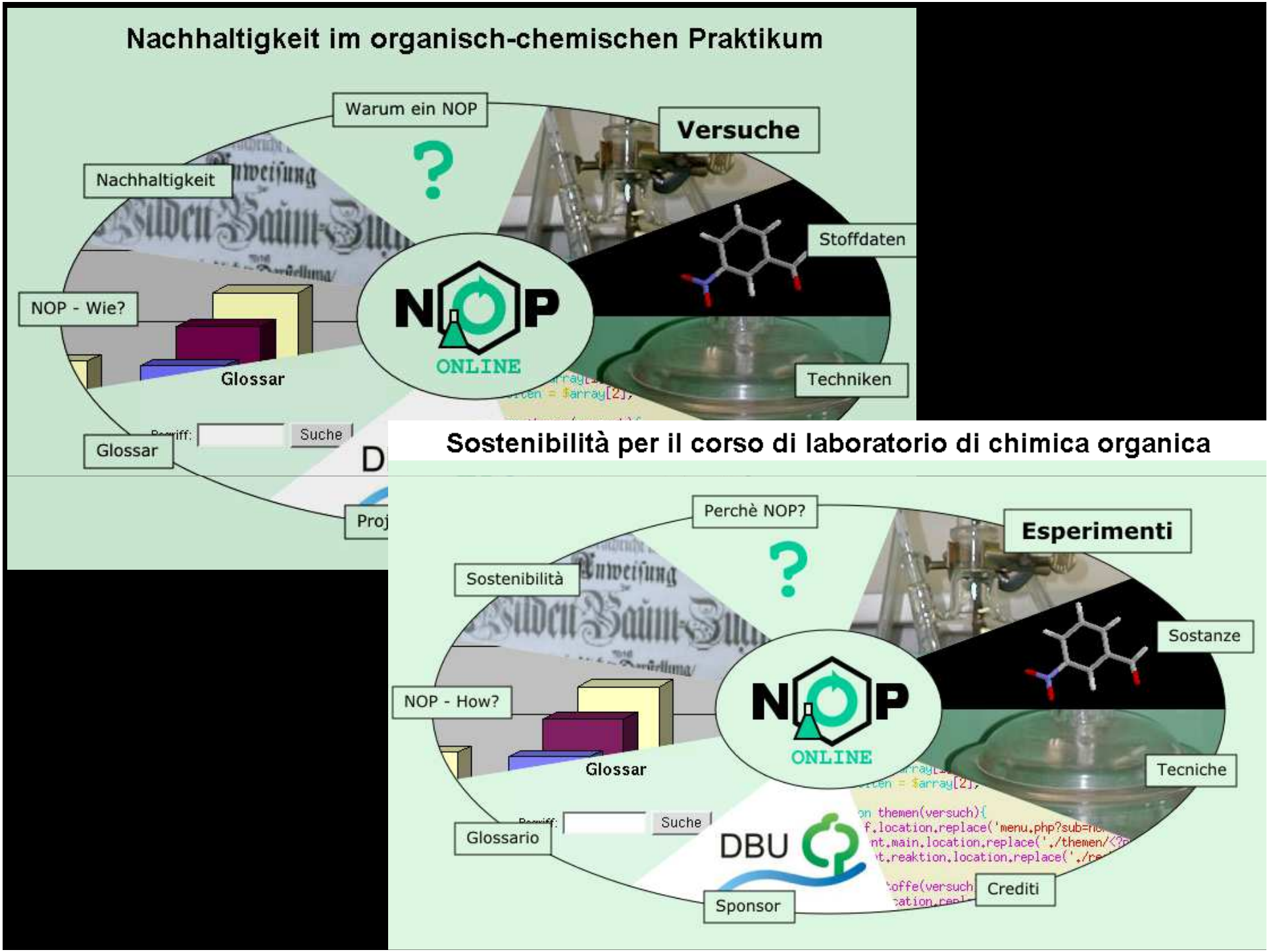
Educt, product and the solvents cyclohexane and tert-butyl methyl ether are biologically not easily degradable, and some are classified as dangerous to the environment because of their toxicity to aquatic organisms.

Summed up we evaluate this experiment with good economic efficiency and acceptable toxicological risks, but a relatively high environmental persistence of the used substances with the "yellow light".

English Change language

pages/experiment.php: March 03, 2006
en/exp_evaluations/html/2003.html: March 03, 2006




Nachhaltigkeit im organisch-chemischen Praktikum
Warum ein NOP
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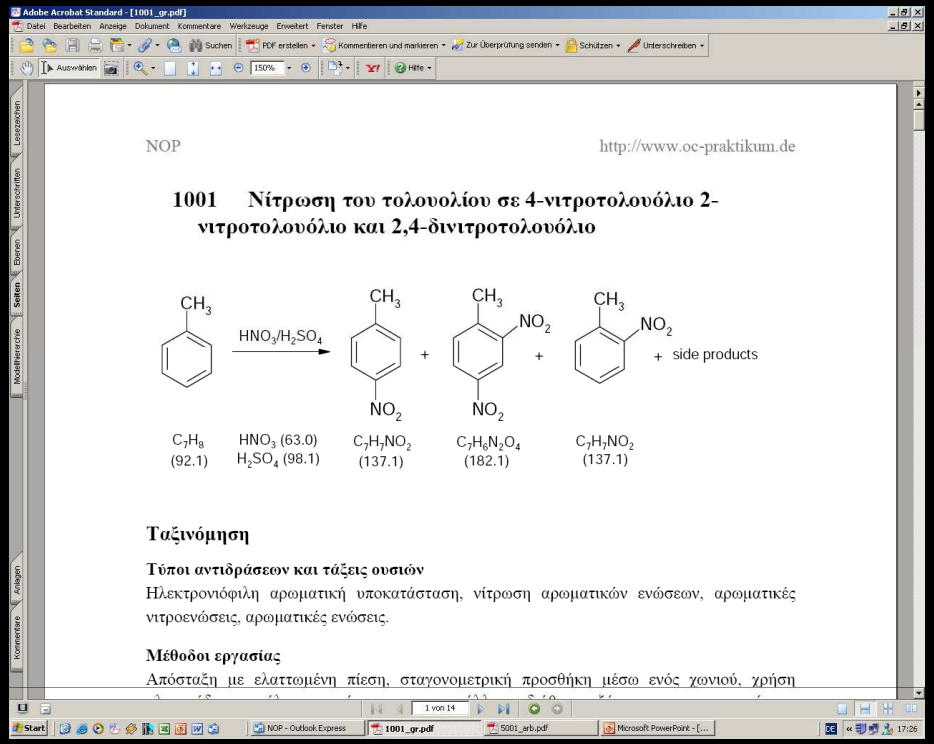


NOP http://www.oc-praktikum.de

1001 Νίτρωση του τολουολίου σε 4-νιτροτολουόλιο 2-νιτροτολουόλιο και 2,4-δινιτροτολουόλιο

C_7H_8 (92.1)	HNO_3 (63.0) H_2SO_4 (98.1)	$C_7H_7NO_2$ (137.1)	$C_7H_6N_2O_4$ (182.1)	$C_7H_7NO_2$ (137.1)

Ταξινόμηση

Τύποι αντιδράσεων και τάξεις ουσιών

Ηλεκτρονιόφιλη αρωματική υποκατάσταση, νίτρωση αρωματικών ενώσεων, αρωματικές νιτροενώσεις, αρωματικές ενώσεις.

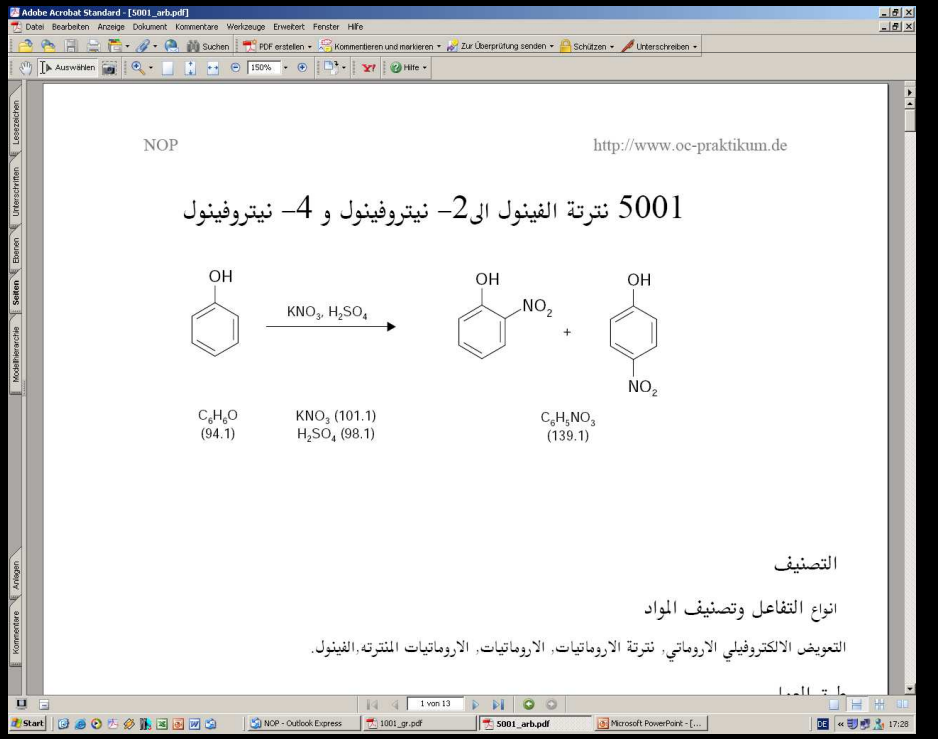
Μέθοδοι εργασίας

Απόσταξη με ελαττωμένη πίεση, σταγονομετρική προσθήκη μέσω ενός χωνιού, χρήση

NOP in Greek

Available Languages

Arabic	Indonesian
Georgian	Italian
German	Portuguese
Greek	Russian
English	Spanish
	Turkish



NOP http://www.oc-praktikum.de

5001 نترتة الفينول الى2– نيتروفينول و 4– نيتروفينول

C_6H_6O (94.1)	KNO_3 (101.1) H_2SO_4 (98.1)	$C_6H_5NO_3$ (139.1)

التصنيف

انواع التفاعل وتصنيف المواد

التعويض الالكتروفيلي الاروماتي، نترتة الاروماتيات، الاروماتيات، الاروماتيات المنترته،الفينول.

NOP in Arabic

NOP - Sustainable Organic Chemistry Lab Course
International Partners

Project granting

Development of the database for

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(German Environmental Foundation)

UMWELT
STIFTUNG