# Data Compilation, Selection, and Derivation of PNEC Values for the Soil Compartment

The Existing Substances Risk Assessment of Nickel was completed in 2008. The straightforward explanation of the goal of this exercise was to determine if the ongoing production and use of nickel in the European Union (EU) causes risks to humans or the environment. The European Union launched the Existing Substances regulation in 2001 to comply with Council Regulation (EEC) 793/93. "Existing" substances were defined as chemical substances in use within the European Community before September 1981 and listed in the European Inventory of Existing Commercial Chemical Substances. Council Regulation (EEC) 793/931 provides a systematic framework for the evaluation of the risks of existing substances to human health and the environment.

The conceptual approach to conducting the environment section of the EU Risk Assessment of Nickel included the following steps (Figure 1):

Field trials for nickel ecotoxicity testing in soil.

- Emissions of nickel and nickel compounds to the environment were quantified for the whole life cycle, i.e., from production, use, and disposal;
- Concentrations of nickel resulting from these emissions were determined in relevant environmental media (water, sediment, soil, tissue) at local and regional scales (PECs);
- Critical effects concentrations (PNECs) were determined for each of the relevant environmental media;
- Exposure concentrations were compared to critical effects concentrations for each of the relevant environmental media (risk characterization); and
- Appropriate corrective actions (also described as risk management) were identified for situations where exposure concentrations were greater than critical effects concentrations. Where exposure concentrations were below critical effects concentrations, there was no need for concern or action.

The EU Risk Assessments for Nickel and Nickel Compounds were developed over the period from 2002 to 2008. The Danish Environmental Protection Agency (DEPA) acted as the Rapporteur in this process, in close collaboration with the international nickel industry. EU Risk Assessment Reports (RARs) for the environment for nickel substances (metallic nickel, nickel carbonate, nickel chloride, nickel nitrate, and nickel sulfate) were submitted in the spring of 2008 after thorough review by the Technical Committee on New and Existing Substances (TCNES), which was comprised of technical representatives from the EU Member States. A final peer review was provided by the Scientific Committee on Health and Environmental Risks (SCHER) (see Section 5). The European Commission's Institute for Health and Consumer Protection published the final Risk Assessment Reports for nickel and nickel compounds in November 2009.

After the EU RARs received approval within Europe, the data sets were discussed at the international level within the Organization of Economic Cooperation and Development (OECD). The nickel ecotoxicity data sets used in the EU RARs were accepted at the OECD's SIDS (Screening Level Information Data Set) Initial Assessment Meeting (SIAM 28, October 2008), as was the use of nickel bioavailability models to normalize the nickel ecotoxicity data.

## 1 INTRODUCTION

Environmental risks are typically characterized in the risk assessment framework by considering the ratio between exposure concentrations and critical effect concentrations. In OECD countries, critical effect concentrations are based on Predicted No-Effects Concentrations (PNECs), which are typically derived from long-term laboratory-based ecotoxicity tests using well-defined protocols on a limited number of species. Such information is usually retrieved from relevant literature and/or internationally recognized databases. Because the quality of the extracted data may vary considerably among individual source documents, it is important to evaluate all ecotoxicity data with regard to their adequacy for PNEC derivation and risk assessment. This fact sheet provides clear guidance on how to perform such evaluation for the soil compartment, including criteria for acceptance (or rejection) of a study in accordance with the purpose of the assessment and examples how these data were applied in the European Union Environmental Risk Assessment for Nickel and Nickel Compounds (EU RA).

In the EU RA, a stepwise approach is used for the derivation of the soil PNEC value. Figure 2 provides an overview of the steps that need to be accomplished in order to derive the critical effect concentrations (PNEC) for nickel for the soil compartment.

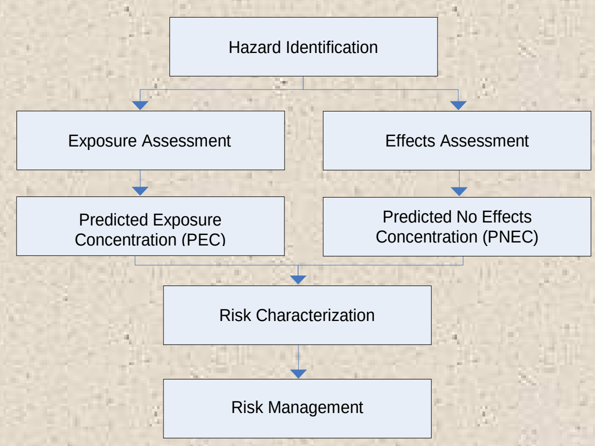


**Figure 1:** Schematic overview of the steps in the EU Environmental Risk Assessment

# 2 GUIDANCE

## 2.1 DATA COMPILATION

The data on the toxicity of nickel to soil organisms were compiled from three main sources: open literature, internationally recognized databases (*e.g.*, Science Direct, Web of Science), and industry-sponsored research programs. A large dataset on the ecotoxicity of nickel to soil organisms was compiled. All gathered data were further screened using the criteria as outlined in Section 2.2.

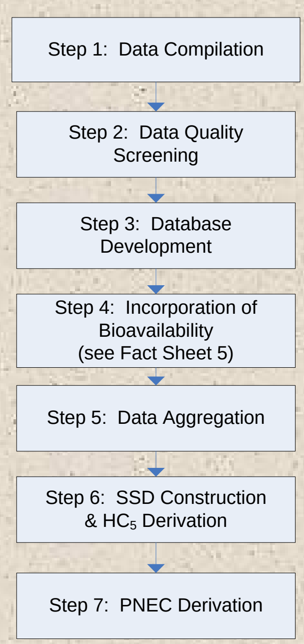


**Figure 2:** Stepwise approach used for the derivation of the soil PNEC value

## 2.2 DATA QUALITY SCREENING

Each individual ecotoxicity data point was screened for quality before incorporation in the nickel ecotoxicity database based on the following criteria[i]:

- the data were retained for the following groups of organisms: higher plants, invertebrates, and micro-organisms;
- the data on soil higher plants & invertebrate organisms covered the following relevant endpoints: survival, growth, reproduction, litter breakdown, and abundance;
- the data on soil micro-organisms covered the following relevant endpoints: respiration, nitrification, mineralization, growth, and enzyme activity;
- toxicity tests for plants, invertebrates, and microbial processes were conducted in natural and artificial soils only, and tests performed under hydroponic conditions were rejected;
- toxicity tests for microbial enzyme activity were rejected if the pH of the test was substantially different from the pH of the undisturbed soil;
- the results reported measured pH and cation exchange capacity (CEC);
- Nickel-only exposure data were considered relevant (studies were rejected if indications of impurities or other substances might have an effect on the toxic properties of nickel);
- the range of the physico-chemistry of the test media (pH and CEC) were within the range of the developed/validated soil regression models (see Fact Sheet 5);
- the tests were performed according to approved international standard test guidelines; however, data from non-standardized tests were also assessed;
- only long-term or chronic toxicity data, involving endpoints that are realized over periods of several days to years depending on the organism, were used;
- the tests were performed according to standard operational procedures, with a detailed description of the methods employed during toxicity testing;
- the toxicity tests were performed with soluble nickel salts (*e.g.*, $NiCl_2$ and $NiSO_4$);
- preference was clearly given on the use of measured nickel concentrations in the test concentrations;
- the toxicity data were related to the total concentration of nickel in soils (defined as strong acid extraction without further confinement) and the test results were expressed as mg Ni/kg dry weight;
- a clear concentration-response was observed;
- toxicity threshold values calculated as $L(E)C_{10}$ (the concentration that causes 10% effect during a specified time interval) values were preferred; however, NOEC values (No Observed Effect Concentration) were also seen as equivalent; and,
- ecotoxicity threshold values data were derived using the proper statistical method.

Only the identified ecotoxicity data fulfilling the above mentioned criteria were used for the soil PNEC derivation.

## 2.3 DATABASE DEVELOPMENT

Applying the above mentioned quality screening criteria to the identified ecotoxicity data resulted in the selection of an extensive high quality database on the ecotoxicity of nickel to soil organisms. Indeed, the Nickel database contains a total of 42 different “species/process mean” values. This includes 52 individual $EC_{10}$/NOEC values for the microbial processes, 16 individual $EC_{10}$/NOEC values for enzymatic processes, 68 individual NOEC values for the plants, and 37 individual values for the invertebrates.

An overview of all accepted individual high quality chronic ecotoxicity data is presented in the Environmental Risk Assessment of Nickel and Nickel Compounds (see Section 5).

## 2.4 INCORPORATION OF BIOAVAILABILITY (DATA NORMALIZATION)

When considering the bioavailability of nickel in soils, different factors are important in determining the ecotoxicity of nickel to soil organisms:

- *Nickel-form:* Nickel can enter the soil environment as soluble, associated with a high bioavailability, or as sparingly soluble, associated with a low bioavailability species.
- *Ageing:* The larger toxicity of nickel in spiked soils compared to corresponding field contaminated soils is highly dependent on the time between the addition of soluble nickel to soils and the measurement of toxicity. The bioavailability and toxicity of nickel in spiked soils tend to decrease with time in a manner that is dependent on soil pH.
- *Normalization:* The toxicity of nickel is highly dependent on soil type. Specifically, nickel toxicity to plants, invertebrates, and microbial processes decreases as the CEC of the soil increases.

For further guidance, see Fact Sheet 5 on bioavailability models for the soil compartment.

## 2.5 DATA AGGREGATION

Aged and/or normalized high quality ecotoxicity data are grouped/aggregated in order to avoid over representation of ecotoxicological data from one particular species/process. The following major rules were used to aggregate data:

- If several chronic NOEC/L(E)$C_{10}$ values based on the same toxicological endpoint were available for a given species, the values were averaged by calculating the geometric mean, resulting in the "species mean" NOEC/L(E)$C_{10}$.
- If several (geometric mean) chronic NOEC/L(E)$C_{10}$ values based on different toxicological endpoints were available for a given species, the lowest (geometric value) value was selected.

After the data aggregation step, only one ecotoxicity value (*i.e.*, the geometric mean for the most sensitive endpoint) was assigned to a particular species.

## 2.6 CALCULATION OF PNEC USING STATISTICAL EXTRAPOLATION METHODS

### Estimation of the $HC_5$ from the species sensitivity distribution

When a large data set for different taxonomic groups is available, the PNEC can be calculated using a statistical extrapolation method. In this approach, the ecotoxicity data were ranked from low (most sensitive species/process) to high (least sensitive species/process). A species sensitivity distribution (SSD) was then constructed by applying an appropriate curve fitting distribution, usually a log-normal distribution, to the (aged and normalized) high quality aggregated chronic toxicity data (Aldenberg & Jaworska, 2000). From the SSD, a 5th percentile value (at the median confidence interval) is calculated (*i.e.*, median $HC_5$) using the software program ETx as described by Van Vlaardingen *et al.* (2004).

### Selection of appropriate assessment factor and derivation of the PNEC

To account for uncertainty, an assessment factor (AF) may be applied to the median $HC_5$. In general, such AFs vary between 1 and 5 and are determined on a case-by-case basis. The soil PNEC would therefore be calculated as follows:

$$\text{soil PNEC} = \text{median } HC_5/AF$$

Based on the available chronic NOEC/L(E)$C_{10}$ data, the following points were considered when determining the AF:

- The overall quality of the database and the endpoints covered (*e.g.*, are all the compiled data representative of "true" chronic exposure?)
- The diversity of the taxonomic groups (Table 1) covered by the database [*e.g.*, do the databases contain, at a minimum, organisms belonging to the six taxonomic groups as defined by the MERAG (Metals Environmental Risk Assessment Guidance) document (2006)?]
- The number of species (*e.g.*, does the SSD cover at least 10 different L(E)$C_{10}$/NOECs and preferably more than 15?)
- Use of bioavailability models and approach for bioavailability correction [*e.g.*, do the bioavailability models (see Fact Sheet 5) allow the toxicity data for all species to be corrected for to ageing/normalizing?]
- Statistical extrapolation (*e.g.*, how well does the SSD fit the toxicity data?)
- Comparisons between field and mesocosm studies and the PNEC (*e.g.*, is the PNEC value protective for the effects observed in mesocosm/field studies?)

In the Nickel EU RA, no mesocosm/field data were available that allowed the determination of threshold concentrations of nickel in soils under field conditions. In addition, increased uncertainty was attributed to the limited number of species and life-strategies for which the bioavailability models have been developed and validated. All other identified criteria were fulfilled. Therefore, based on the weight of evidence, the Danish Rapporteur proposed to use an AF of 2.

| *Taxonomic Groups* |
|---|
| 1. Microbe mediated processes (*e.g.*, respiration, denitrification, N- mineralization, etc.) |
| 2. An insect (*e.g.,* Collembola) |
| 3. An oligochaete (*e.g.,* Eisenia, Enchytreus) |
| 4. A family in any order of oligochaete or any phylum not already presented |
| 5. Higher plants (monocotyle) |
| 6. Higher plants (dicotyle) |

**Table 1:** Taxonomic group requirements according to the Criteria developed in the MERAG document (ICMM, 2007)

# 3 EXAMPLE

## 3.1 DATA COMPILATION

See Section 2.1

## 3.2 DATA QUALITY SCREENING

The quality screening criteria as defined in Section 2.2 were applied to select the high quality chronic ecotoxicity data of nickel to soil organisms.

## 3.3 DATABASE DEVELOPMENT

An overview of all accepted individual high quality chronic ecotoxicity data is presented in the Environmental Risk Assessment of Nickel and Nickel Compounds (see Section 5).

## 3.4 DATA NORMALIZATION

In this example, the data were normalized to the physico-chemical conditions prevailing in the Danish soils using the ageing and bioavailability models as explained in Fact Sheet 5.

| *Taxonomic Group/Process* | *Species* | *Most Sensitive Endpoint* | *Species Mean NOEC/L(E)C$_{10}$ Value (mg/kg)* |
|---|---|---|---|
| Higher plants | *Lycopersicon esculentum* | Shoot yield | 182 |
| | *Hordeum vulgare* | Root yield | 293 |
| | *Spinacia oleracea* | Total yield | 75 |
| | *Avena sativa* | Yield grain | 82 |
| | *Medicago sativa* | Yield total plant | 105 |
| | *Raphanus sativus* | Yield | 170 |
| | *Allium cepa* | Yield | 277 |
| | *Trigonella poenumgraceum* | Yield | 497 |
| | *Lolium perenne* | Yield | 46 |
| | *Lactuca sativa* | Yield leaves | 139 |
| | *Zea mays* | Not reported | 209 |
| Invertebrates | *Folsomia candida* | Reproduction | 398 |
| | *Folsomia fimetaria* | Reproduction | 277 |
| | *Eisenia fetida* | Reproduction | 281 |
| | *Enchytraeus albidus* | Reproduction | 164 |
| | *Eisenia veneta* | Reproduction | 132 |
| | *Lumbricus rubellus* | Mortality | 918 |
| Microbial process/species | Nitrification | $NO_3$ production | 160 |
| | N-mineralisation | $NO_3$ production | 253 |
| | Glucose respiration | $CO_2$ release | 168 |
| | Maize respiration | $CO_2$ release | 255 |
| | *Aspergillus flavipes* | Hyphal growth | 492 |
| | *Aspergillus flavus* | Hyphal growth | 554 |
| | *Aspergillus clavatus* | Hyphal growth | 43 |
| | *Aspergillus niger* | Hyphal growth | 563 |
| | *Penicillium vermiculatum* | Hyphal growth | 163 |
| | *Rhizopus stolonifer* | Hyphal growth | 412 |
| | *Trichoderma viride* | Hyphal growth | 738 |
| | *Gliocladium sp.* | Hyphal growth | 294 |
| | *Serratia marcescens* | Colony count | 234 |
| | *Proteus vulgaris* | Colony count | 46 |
| | *Bacillus cereus* | Colony count | 408 |
| | *Nocardia rhodochrous* | Colony count | 263 |
| | *Rhodotorula rubra* | Colony count | 357 |
| | Respiration | $CO_2$ release | 137 |
| | Glutamate respiration | $CO_2$ release | 51 |
| | ATP content | / | 87 |
| Enzymatic activity | Urease | / | 293 |
| | Phosphatase | / | 639 |
| | Arylsulfatase | / | 1164 |
| | Dehydrogenase | / | 28 |
| | Saccharase | / | 87 |
| | Protease | / | 87 |

**Table 2:** Selected species mean ecotoxicity data to nickel for the most sensitive endpoint

The soils in Denmark are characterized by a pH of 6.3, a CEC of 10.4 cmol/kg, a clay content of 8.9%, and organic matter (OM) content of 0.6%.

## 3.5 DATA AGGREGATION

The selected normalized individual high quality chronic ecotoxicity data of nickel to soil organisms are aggregated according to the criteria mentioned in Section 2.5. An overview of the aged normalized (to the conditions prevailing in the Danish soils) species mean NOEC/L(E)$C_{10}$ values for the most sensitive endpoint is provided in Table 2.

## 3.6 SSD CONSTRUCTION AND MEDIAN $HC_5$ DERIVATION

The species mean NOEC/L(E)$C_{10}$ values in Table 2 were further ranked from low to high. Subsequently, a log-normal distribution was fitted through the ranked species mean toxicity data. From this SSD the median 5$^{th}$ percentile was calculated using the ETx model. The SSD and the median $HC_5$ value for the normalized ecotoxicity data to the physico-chemical conditions prevailing in the soils in Denmark are presented in Figure 3.

## 3.7 PNEC DERIVATION

An AF of 2 is applied to the median $HC_5$ value resulting in a soil PNEC = median $HC_5$ / 2 = 47.0 mg Ni/kg / 2 = 23.5 mg Ni/kg.

The example of the SSD construction and PNEC derivation for nickel, as shown above, only applies for the soil chemistry prevailing in the Danish soils. However, other soil chemistries are encountered in the EU soils resulting, therefore, in the setting of different PNEC values for Ni. These different eco-regions, as shown in Table 3, have been selected in the EU Risk Assessment to provide *examples* of typical conditions covering a wide range of physico-chemical conditions (pH between 3.0 and 7.5, CEC between 2.4 and 36 cmol/kg, clay between 7 and 46%) occurring in EU soils. Therefore, PNEC values for typical eco-regions in EU soils vary, depending on the soil chemistry, between 4.3 and 96.2 mg Ni/kg. The soil chemistry and median $HC_5$/PNEC values calculated for the different selected eco-regions in EU soils are summarized in Table 3.

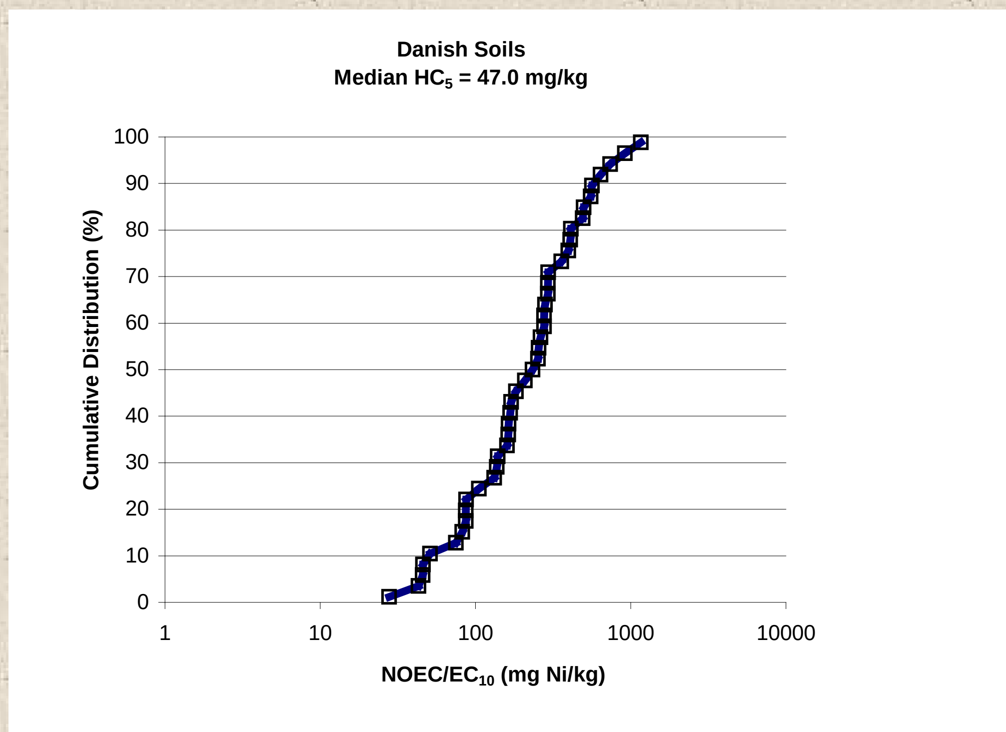


**Figure 3:** SSD and median $HC_5$ derivation for nickel using normalized ecotoxicity data for the Danish soils

| Eco-Region | Soil Use | Soil Chemistry | L/A Factor[ii] | Median $HC_5$ (µg/L) | PNEC (µg/L)[iii] |
|---|---|---|---|---|---|
| Acid sandy soil in Sweden | Arable land | pH 4.8, OM 2.8%, clay 7%, CEC 2.4 cmol/kg | 1.05 | 8.5 | 4.3 |
| Loamy soil in The Netherlands | Arable land | pH 7.5, OM 2.2%, clay 26%, CEC 20 cmol/kg | 3.01 | 99.2 | 49.6 |
| Peaty soil in The Netherlands | Grassland | pH 4.7, OM 40%, clay 24%, CEC 35 cmol/kg | 1.04 | 186.3 | 93.2 |
| Acid sandy soil in Germany | Forest land | pH 3.0, OM 9%, clay 7%, CEC 6 cmol/kg | 1.0 | 25.0 | 12.5 |
| Clay soil in Greece | Woodland | pH 7.4, OM 4.5%, clay 46%, CEC 36 cmol/kg | 2.75 | 192.3 | 96.2 |
| Soils of different types in Denmark | Arable & forest land | pH 6.3, OM 0.6%, clay 8.9%, CEC 10.4 cmol/kg | 1.38 | 47.1 | 23.6 |

**Table 3:** Overview of the soil chemistry and median $HC_5$/PNEC values for the different selected EU eco-regions

## 4 CONCLUSIONS AND NEXT STEPS IN RA

This fact sheet presents the approach for data gathering, data selection, and data aggregation to be used for derivation of the PNEC value for the soil environment based on the SSD. Because the ecotoxicity of nickel is mitigated by the long-term reactions with the soil (*i.e.*, ageing) and the physico-chemistry of the soils (*i.e.*, pH, clay content, organic matter content), it is highly recommended to age and normalize the ecotoxicity data for PNEC derivation using the available bioavailability models as described in the Fact Sheet 5.

## 5 LINK TO EU RISK ASSESSMENT DOCUMENTS

The final report on the Environmental Risk assessment of Nickel and Nickel Compounds can be retrieved from the following website:

http://esis.jrc.ec.europa.eu/doc/risk_assessment/REPORT/nickelreport311.pdf
(last accessed July 2015)

The opinion of the SCHER can be found at the following address:

http://ec.europa.eu/health/ph_risk/committees/04_scher/docs/scher_o_112.pdf
(last accessed July 2015)

## 6 REFERENCES

Aldenberg, T. and Jaworska, J. S. 2000. Estimation of the hazardous concentration and fraction affected for normally distributed species sensitivity distributions. Ecotoxicology and Environmental Safety, 46, 1-18.

ICMM. 2007. MERAG: Metals Environmental Risk Assessment Guidance. ISBN 978-0-9553591-2-5.

SIDS (Screening Level Information Data Set) Initial Assessment Meeting (SIAM) 28. Ottawa, Ontario, Canada. October, 2008.

Van Vlaardingen, P. L.; T. P. Traas; A. M. Wintersen; and T. Aldenberg. 2004. ETX 2.0. A program to calculate risk limits and fraction affected, based on normal species sensitivity distributions. Report no. 601501028/2004, National Institute for Public Health and the Environment (RIVM), Bilthoven, The Netherlands.

i The application of the quality screening criteria would also apply in case additional or new ecotoxicity data would be considered.

ii The term leaching-ageing factor (L/A actor) refers to the combined effect of leaching (due to changing ionic strength) and ageing (due to long-term reactions) on nickel bioavailability and toxicity in soil.

iii PNEC is calculated using an AF of 2.

## Fact Sheets on the European Union Environmental Risk Assessment of Nickel

This is the second in a series of fact sheets addressing issues specific to the environment section of the European Union's Existing Substances Risk Assessment of Nickel (EU RA). The fact sheets are intended to assist the reader in understanding the complex environmental issues and concepts presented in the EU RA by summarizing key technical information and providing guidance for implementation.

NiPERA welcomes questions about the concepts and approaches implemented in the EU RA. For inquiries, please contact:

NiPERA, Inc.
2525 Meridian Parkway, Suite 240
Durham, NC 27713, USA
Telephone: 1-919-595-1950

Chris Schlekat, Ph.D., DABT
cschlekat@nipera.org

Emily Garman, Ph.D.
egarman@nipera.org

This fact sheet was prepared by Patrick Van Sprang of ARCHE, Stapelplein 70, b 104, B-9000 Gent, Belgium.
patrick.vansprang@arche-consulting.be