

## Optimization potentials of laboratory reactions -

### Basic rules for sustainable syntheses

In course of investigating some reactions of the NOP some general weak points could be identified. These affect particularly the energy as well as the resource consumption of laboratory reactions. By means of some "simple" actions the recognized problems can be reduced or avoided during reaction planning. For this purpose the following "basic rules for sustainable syntheses" were created [1]. The rules can contribute to a holistic thinking, i.e. including preliminary and posterior processes, and for improving the sustainability of a reaction from an ecological point of view.

#### Basic rules for sustainable syntheses – minimizing energy consumption

The environmental impact of laboratory reactions is affected substantially by the electricity consumed during the experimental process and the environmental effects caused by supplying that energy. The high energy consumption of the examined reactions has its cause predominantly in high energy loss into the environment during the synthesis. In the following "basic rules for sustainable syntheses" are defined, whose application leads to a reduction of energy loss within laboratory reactions.

<i>thermal insulations</i>	The experimental set up has to be isolated in the best possible way.
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#### *basic rule 1: thermal conditions*

The heat transfer coefficient (k-value) can be reduced by insulation. This leads to a smaller heat transfer and thus to reduced energy loss. This loss can be decreased further by making the temperature gradient among reaction medium and environment smaller. Since the energy loss is also time-dependent, the duration of the experiment has a large part in its minimization. For this reason an exact knowledge of the reaction process is essential.



*experimental conditions* The reaction temperature as well as the duration should be limited to the necessary degree.

*basic rule 2: experimental conditions*

Another parameter affecting energy loss is the surface of the equipment, which separates the reaction mixture against the environment. The smaller the surface, the smaller the energy loss by heat conduction or heat radiation. The ratio of surface and volume should be chosen wisely.

*choice of equipment* The experimental set up should be adapted to the scheduled quantity.

*basic rule 3: choice of equipment*

The differences in energy consumption encountered with the different methods of energy input can be traced to the two basic rules already drafted - insulation and experimental conditions. Since complete insulation of the heating medium is usually not possible, favorable methods of energy input should be preferred.

*energy input* The use of a heating mantle is to be preferred to an heating stage with an oil bath. New methods of energy input (e.g. microwave or ultrasonic) can have positive effects.

*basic rule 4: energy input*

Apart from high energy loss by transferring thermal energy to the environment, there are other factors contributing to the energy consumption of laboratory syntheses. Thus auxiliary materials affect the energy needed for heating and keeping the reaction temperature.

*c<sub>p</sub> auxiliary materials* Auxiliary materials should feature a specific heat capacity as small as possible

*basic rule 5: specific heat capacity of auxiliary materials*



Among the auxiliary materials special attention has to be paid to the solvents, since they usually make up a large part of the reaction mixture. For this reason the requirements to solvents are put in their own basic rule.

*c<sub>p</sub> solvent*

Solvents are to be selected such that they have a small specific heat capacity and a low necessary reaction temperature.

*basic rule 6: specific heat capacity of the solvent*

The use of auxiliary materials can be optimized with respect to energy consumption in still another way: The correlation of resource consumption and energy consumption is very important.

### **Basic rules for sustainable syntheses – minimizing energy and resource consumption**

Extensive use of auxiliary materials within reaction and processing affects resource consumption negatively. Furthermore the quantity of auxiliary materials used contributes to the energy consumption – for example by requiring additional energy for heating. This affects resource and energy consumption in the preproduction, which is important from an holistic view. Often auxiliary materials are used far in excess.

*quantity of auxiliary materials*

The auxiliary material quantity is to be reduced to the materials amount really needed. Preliminary experiments help to determine the correct amount. If possible the removal of auxiliary materials is to be integrated into the reaction.

*basic rule 7: quantity of auxiliary materials*



The cooling water is of big importance – similar to the solvent – due to its portion of the material consumption.

**cooling water**

The cooling water flow is to be adjusted to the necessary minimum. The use of a cooling water cycle can affect the environmental impact of a reaction favourably.

*basic rule 8: cooling water*

Cooling water consumption can be reduced by using a cooling circuit or a cryostat, which however increases energy consumption. If ice is needed for cooling reactions, its use is to be limited likewise due to the high energy consumption required for its production to the necessary quantity.

**ice**

Ice for cooling the reaction is to be used with consideration to reaction security as economically as possible.

*basic rule 9: ice*

Apart from the quantity of used materials their characteristics have also a definite influence on the environmental impact of laboratory syntheses. In the following general basic rules for the minimization of environmental impacts are formulated. The interest is focused on auxiliary materials.

**Basic rules for sustainable syntheses - minimizing toxicity potentials**

While creating optimization rules for minimizing toxicity potentials of educts are not taken into account. An optimization towards generally non-toxic raw materials would be desirable, but a variation of the educts for the synthesis of a certain compound is not always possible. The problems with selecting the used auxiliary materials are less significant. Here a consideration of the substantial effect potentials seems more simply.

*toxicity of solvents* While selecting solvents also their toxicity potential is to be considered. The substances must not reach the environment.

*basic rule 10: toxicity of solvents*

*toxicity of auxiliary materials* While selecting auxiliary materials their toxicity potential is to materials be considered. After use these materials must be recycled or disposed environmentally benign.

*basic rule 11: toxicity of auxiliary materials*

As mentioned in previous chapters, high environmental impacts can originate from preliminary and posterior processes of the actual reaction, too. A consideration of the preproduction of the assigned educts and auxiliary materials can help to reduce the environmental impact from a holistic point of view.

### **Basic rules for sustainable syntheses - consideration of preproduction**

Within the production of chemicals often different environmental impacts are caused depending upon complexity of the molecule. Very often, these environmental impacts are not influenceable. However, the experimenter can reduce the insertion of chemicals to the amount necessary for the reaction. The efforts to provide material flow balances for different chemicals have increased in the past years. Thus a consideration of the preproduction of educts and auxiliary materials will become more simple in the future. An optimization of reactions is possible by the selection of materials with small environmental impact in the preproduction.



*preproduction*      The loads from educts and auxiliary materials in preliminary processes are to be considered while selecting the material.

*basic rule 12: consideration of preliminary processes*

In following table (Tab. 1) all "basic rules for sustainable syntheses" are presented again and summarized. Additionally, each basic rule is provided with a pictogram that shall simplify its application, similarly as with **symbols of danger**.

The consideration of these optimization rules can help to reduce the energy and resource consumption from laboratory reactions as well as their environmental impacts. Furthermore, future decision makers can already be sensitized for the interests of a sustainable development by a consistent application of the "basic rules for sustainable syntheses" in the training.



	thermal insulation	The experimental set up have to be isolated in the best possible way.
	experimental conditions	The reaction temperature as well as the duration should be limited to the necessary degree.
	choice of equipment	The experimental set up should be adapted to the scheduled quantity.
	energy input	The use of heating mantles is to be preferred to a heating stage with an oil bath. New methods of energy input (e.g. microwave or ultrasonic) can have positive effects.
	$c_p$ auxiliary materials	Auxiliary materials should feature a specific heat capacity as small as possible.
	$c_p$ solvent	Solvents are to be selected such that they have a small specific heat capacity and a low necessary reaction temperature.
	quantity of auxiliary materials	The auxiliary material quantity is to be reduced to the amount really needed. Preliminary experiments help to determine the correct amount. If possible the removal of auxiliary materials is to be integrated into the reaction.
	cooling water	The cooling water flow is to be adjusted to the necessary minimum. The use of a cooling water cycle can affect the environmental impact of a reaction favourably.
	ice	Ice for cooling the reaction is to be used with consideration to reaction security as economically as possible.
	toxicity of solvents	While selecting solvents also their toxicity potential is to be considered. The substances must not reach the environment.
	toxicity of auxiliary materials	While selecting auxiliary materials their toxicity potential is to be considered. After use these materials must be recycled or disposed environmentally benign.
	preproduction	The loads from educts and auxiliary materials preliminary processes are to be considered while selecting the material.

*tab.1: basic rules for lasting syntheses*

[1] Diehlmann, A., Kreisel, G., Gorges, R. (2003). Contribution to "Developing Sustainability" in Chemical Education. *The Chemical Educator*, 8