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Screening in electrosynthesis

Rapid and sustainable development of tomorrow's innovative chemistry

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The screening system described here accelerates and simplifies the investigation and optimization of new electrochemical synthesis steps on a resource-efficient scale and thus contributes significantly to the establishment of electrosynthesis methods.

Electrosynthesis in the 21st century

Organic electrochemistry has its origins in the 19th century [2]. However, due to the increasing social weight of the approaching climate catastrophe and the enormous urgency to counteract it, it is experiencing a surprisingly pronounced renaissance and has become an important method of the 21st century [3]. Conventional synthesis methods often require the use of heavy metals as well as other harmful chemicals, whereas great efforts are being made to develop metal-free and sustainable electrochemical methods [4,5,6]. Organic electrosynthesis thus provides answers to many problems faced by chemists and represents a strategically important alternative to conventional methods [7]. With careful planning, electrochemistry can produce sustainable processes in the spirit of green chemistry [8]. In this context, electrons serve as a clean reagent, which can efficiently avoid reagent waste [9]. Special attention is also paid to the use of renewable raw materials [8,10]. The rapid progress in this field is made possible by appropriately optimized workflows in the screening phase and the use of suitable apparatus. Small batch sizes and a high degree of parallelization enable time- and cost-efficient research [1].

Electrosynthesis shortens processes, saves energy and reduces environmental impact. The IKA Screening System is perfectly suited for constant current electrosynthesis in „multibatch" mode. The combination of both divided and undivided batch cells enables you to quickly carry out research on multiple electroconversions at the same time. More information can be found on the IKA website.

Electrosynthetic screening

An industrial process as well as any new chemical synthesis is usually accompanied by intensive research and optimization [11]. This procedure requires a large number of experiments to be carried out and is therefore usually time-consuming and expensive. Accordingly, these experiments are commonly performed on a small, resource-efficient screening scale. In a subsequent scale-up, the results of the screening experiments can be validated. In order to achieve the highest possible reproducibility of the results of electrosyntheses, it is necessary to create and adhere to

standards in the performance and description of the experiments [12]. An important basis for this is a uniform experimental setup that meets the special requirements of electrochemical screening [1].

Electrosynthetic screening has developed into a fundamental method for the study of new reactions and is thus the basis of many discoveries in recent electrosynthesis. For example, the study of a large number of modern anodic homo- and cross-coupling reactions was massively accelerated by this electrochemical screening [5,6]. Within a few years, a large number of different substrates were made accessible for this reaction (fig. 1). In 2006, this conversion was first investigated using the homocoupling of 2,4-dimethylphenol [13]. This was followed by several other phenols [14] and in 2010 the first anodic cross-coupling of phenols with different arenes was found [15]. After, some phenol-phenol cross-couplings, first completely unprotected [16]- and later with the aid of protecting groups [17], as well as the synthesis of more complex target structures under double cross-coupling [18] - were followed by the exploitation of anilines [19] and various heterocyclic coupling components [20] for this type of reaction. After the synthesis of para-biphenols [21], the authors even succeeded in introducing phenols with electron-withdrawing substituents into the coupling reaction [22]. Most of the reactions listed in figure 1 (marked in green) were studied using a screening apparatus developed at Mainz University in the AG Waldvogel [1]. The described screening system as well as suitable accessories are currently manufactured and commercially distributed by IKA. This opens up the general entry into electrosynthesis and promotes reproducibility across laboratories.

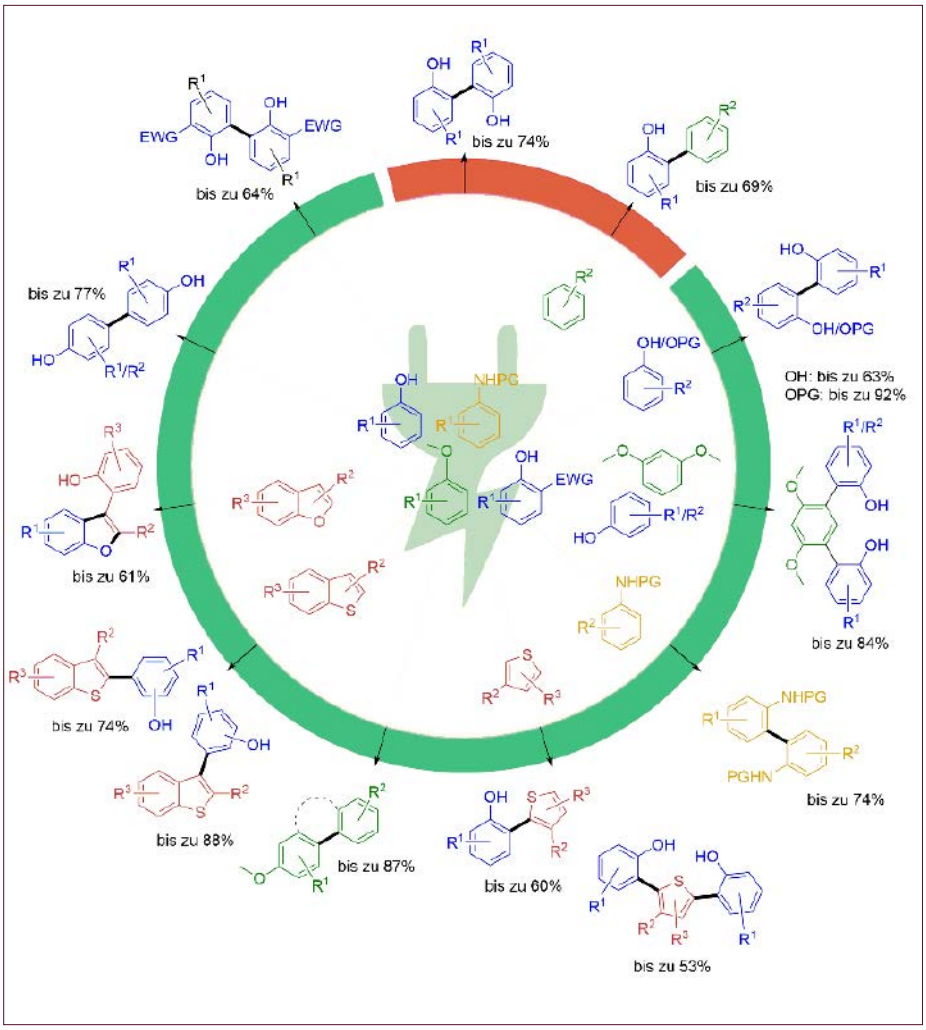

Fig. 1: Selected anodic homo- and cross-coupling reactions. Highlighted in red: before the development of the electrochemical screening system. Labeled green: investigated with the aid of the screening system.

The commercial screening system in detail

This screening system is available for both divided and undivided electrolytic cells and allows 6 or 8 electrolyses to be carried out in parallel on a single magnetic stirring plate. The electrolysis cells can be uniformly tempered with the aid of the heating plate and the manifold, and the electrolysis-specific parameters of current density and voltage can be set separately for each cell due to the multichannel DC sources [23]. Common carbon as well as metal-based electrode materials in the required dimensions are also available, opening up a wide field of potential applications. In addition to this plug and play concept, novel electrode systems can also

Fig. 2: IKA's commercial screening system. Left: Setup consisting of two multi-channel DC power sources, a hot plate and the reaction block including 8 undivided cells. Top right: Reaction block and single divided electrolytic cell plus two half cells. Bottom right: Reaction block and single undivided electrolytic cell.

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Fig. 3: Electrodes made of different materials for the screening system.

be easily tested here in electrosynthesis, as the electrodes used have simple dimensions of 3 mm x 10 mm x 70 mm.

The current sources offered allow electrosynthesis up to very high current densities (>1A/cm2) and can also be used later on to operate larger batch-type electrolysis cells, such as those from Sigma-Aldrich's SynLectro series [24]. In addition, a gas distributor enables reactions under uniform gas flow. On the one hand, this allows gas to be used as a reaction partner but also allows reactions to be carried out under inert conditions, such as exclusion of oxygen. Modified Teflon cells are now also available, which can be equipped with gas diffusion electrodes and thus supply gaseous components to the electrosynthesis even more efficiently.

Fig. 4: Accessories for electrolysis with gaseous components. Left: Gas distributor, for dividing a gas stream among the screening cells. Right: Divided screening cell for gas diffusion electrodes.

Modern screening methods

The speed of establishing electrolysis achieved by parallelization and the small screening scale enables the combination of modern exploration and optimization methods from fields such as design of experiment or machine learning. This linkage is emerging as an extremely powerful tool in the exploration and optimization of new electroorganic reactions. Especially for reactions where the effects of the reaction parameters on the figure of merit interact with each other, optimization sometimes proves to be very complex [25]. Here, the combination of the screening system with the aforementioned advanced methods is largely responsible for the success of the optimization process. This technology represents the backbone of electrosynthesis research in Mainz.

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IKA

IKA Screening System

The IKA Screening System is perfectly suited to galvanostatic electro-synthesis in “multibatch” mode. The combination of both divided and undivided batch cells enables you to carry out research on multiple probes at the same time. In addition, you can combine the system with other equipment in the laboratory

Parallel batch electrosynthesis using parameters predefined by you:

> Up to 8 undivided or 6 divided cells simultaneously
> Synthesize sufficient quantities for GC, LC or NMR analysis processes
> Separate control for each cell
> Easy testing of identical or differing samples
> Fast identification of ideal process parameters
> Digital recording of test parameters
> Simultaneous mixing and heating
> Full temperature control (PT 1000) using heat block
> Control and automation via Labworldsoft 6.0
> Time and resources savings

Screening System Package
(6 divided cells)
Ident. No. 0040003631

Screening System Package
(8 undivided cells)
Ident. No. 0040003642

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