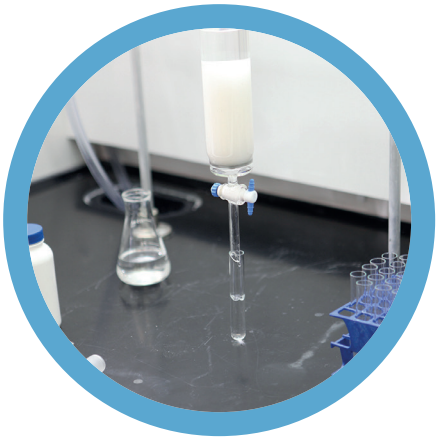
APPLICATION NOTE
#SF002-0

SiliCycle

SiliaFlash®



# The Brockmann & Schodder Activity Test

A common way to validate and compare silica gels with the same particle size distribution is to perform a side-by-side chromatographic separation of a well understood sample mixture. However, for users who do not have access to a known and well characterized sample mixture, the Brockmann & Schodder test can be useful[1]. This test was developed on aluminium oxide but, as mentioned by the authors, it can be adapted to silica.

[1]Brockmann, H. Schodder, H. *Ber. Deut. Chem. Ges*. **1941** (*74*), 73.

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# The Brockmann & Schodder Activity Test

First described by Brockmann & Schodder, the test showed a clear relationship between sorbent water content and its retention characteristics. One of the conclusions drawn at that time, which is still considered to be important by process engineers and chemists, is that reproducible and well controlled water content is mandatory for the achievement of highly reproducible intra and inter-column chromatographic separations.

The effect of water content on a separation can be determined by activating a sorbent through complete dehydration. This is accomplished through heating the silica gel at 110°C for the complete removal of water leading to the most active form of silica (*Grade I on the Brockmann & Schodder test*). Upon activation, the user will adjust the water content by adding water to the sorbent (*Grades II to V are less active; they have a lower adsorption capacity leading to a lower retention capacity*) and validating the impact on the chromatographic performance for the compounds of interest. Interestingly, even if a completely activated (*dried*) silica is considered to have the highest activity, the most active form might not give reproducible and better chromatographic results. Thus, most users prefer to buy silica with already well controlled water content.

The Brockmann & Schodder test is performed by separating mixtures of two dyes varying with the Brockmann grade of activity, thus water content of the sorbent. Due to their coloration visible to the naked eye, it was possible to easily follow their progress on the column. The standard Brockmann & Schodder activity grades are shown in Table 1.

**Table 1**: Standard Brockmann & Schodder activity grades

| Standard Brockmann & Schodder Activity Grades | |
|---|---|
| Water content (%) | Activity grade |
| 0 | I |
| 10 | II |
| 12 | III |
| 15 | IV |
| 20 | V |

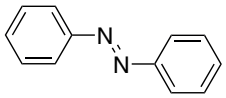
Adsorption capacity

For the evaluation of the chromatographic efficiency, the solvent systems used for elution and dissolution of the dyes consists of benzene / petroleum ether at a 20 / 80 (*v/v*) ratio. The binary dye mixtures used are presented in Table 2.

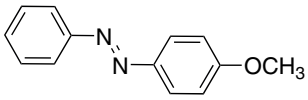
**Table 2**: Binary dye mixtures used for the evaluation of chromatographic efficiency

| Binary Dye Mixtures Used for the Evaluation of Chromatographic Efficiency | | |
|---|---|---|
| Mixture | Compound 1 | Compound 2 |
| 1 | Azobenzene (***AB***) | p-methoxy azobenzene (***MAB***) |
| 2 | p-methoxy azobenzene (***MAB***) | Sudan yellow (***SY***) |
| 3 | Sudan yellow (***SY***) | Sudan red (***SR***) |
| 4 | Sudan red (***SR***) | p-aminoazobenzene (***AAB***) |
| 5 | p-aminoazobenzene (***AAB***) | p-hydroxyazobenzene (***HAB***) |

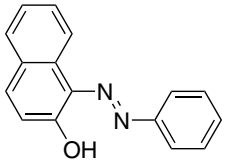
Chemical structure of the dyes:



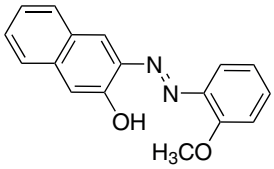
Azobenzene (***AB***)



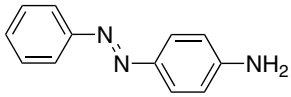
p-methoxy azobenzene (***MAB***)



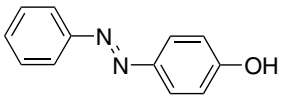
Sudan yellow (***SY***)



Sudan red (***SR***)



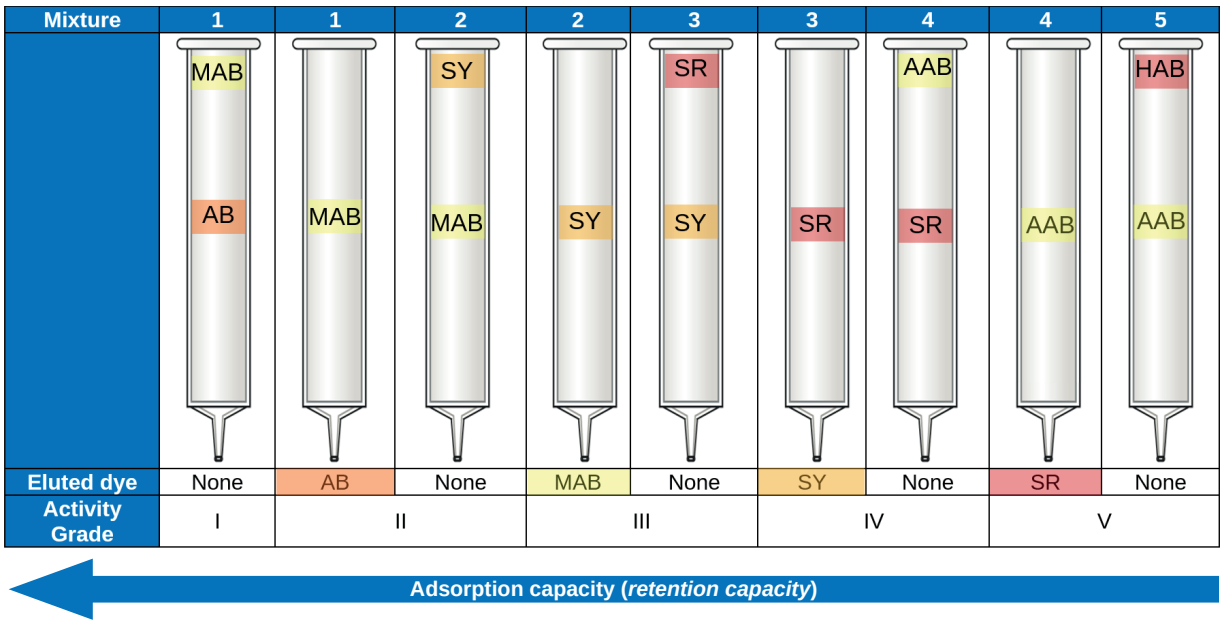
p-aminoazobenzene (***AAB***)



p-hydroxyazobenzene (***HAB***)

To prepare a mixture, 20 mg of each of the two dyes were weighed and dissolved in 50 mL of the solvent (*benzene / petroleum ether at a 20 / 80 (v/v) ratio*). Then, the chromatographic performance of the silica gel was determined by applying 10 mL of a binary dye mixture on a 15 mm diameter column (*packed with exactly 50 mm in height with the sorbent to be tested*). Upon application, elution of the dyes was done using 20 mL of the solvent. As shown on Figure 1, the activity of the silica gel was determined using all the listed (*Table 2*) dyes mixtures.

**Figure 1**: Chromatographic performance according to the activity grade




These experiments clearly demonstrate that the water content of a silica gel has an influence on the chromatographic results obtained and confirm the importance of properly controlling this parameter.

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