13.08.2018

Beginning of the synthesis of the Thiol-Eslicarbazepin in Toluen.
Eslicarbazepin was treated with 0.5 mole equivalent of Lawesson's reagent in Toluene.
Reflux at 120°C gave a yellow liquid with white solids.
Purification via HPLC.
A sample was taken and given to mass spectrometry.

14.08.2018
The MS-Data was examined.
MS-Data gave a yield of < 10%.
Drying of 1,2-dimethoxyethane with CaH_2 overnight.

15.08.2018
Distillation of 1,2-dimethoxyethane, treating with N_2 for inert storage.

16.08.2018
Alternative synthesis of the Thiol-Eslicarbazepin treated with 1,2-dimethoxyethane (DME) at room temperature with 0.5 mole equivalent of Lawesson's reagent, (Method OH->SH).

17.08.2018
Drying the Thiol-Eslicarbazepin and purification by column chromatography (-> methods).
A sample was taken and given to mass spectrometry.

20.08.2018
The MS-Data was examined showed indications of large impurities.
Further purification of the Thiol-Eslicarbazepin by column chromatography.

21.08.2018
First attempt of the synthesis of the azide-Donor (method Azid-D). Sodium azide was suspended in Sulfuryl chloride and stirred overnight.

22.08.2018
The azide mixture was treated with Imidazole and dried in vacuo. After suspending in ethyl acetate the solution was treated with sulfuric acid to yield a crude, yellow solid.
A sample was taken and given to mass spectrometry.

23.08.2018

The MS-Data was showed no yield.
The second attempt for the synthesis of the azide-donor was started.
Sulfuryl chloride was treated with sodium azide and imidazole, suspended in acetonitrile and stirred for three hours. The product was dried in vacuo, solved in ethyl acetate and treated with sulfuric acid. No product was obtained.
The third attempt for the synthesis of the azide-donor was started.
An ice-cooled Suspension of sodium azide in acetonitrile was treated with Sulfuryl chloride.
Synthesis Disulfide-Eslicarbazepine was started.
The synthesis was performed under inert conditions. The Thiol-Eslicarbazepin was solved in THF/H_2O, treated with 2,2'-Dithiodiethylamine and stirred overnight.

24.08.2018
The ice-cooled solution of the azide was treated portion-wise with Imidazole.
It was stirred for three hours, diluted with ethyl acetate, washed, saturated and treated with sulfuric acid. After stirring for one hour the crude, colorless product was obtained. The

product was dried in vacuo. The solution of the S-S-Eslicarbazepine was in a rotary evaporator to obtain a crystalline, colorless solid.

27.08.2018
The S-S-Eslicarbazepine was further purified by washing with ethyl acetate.
A sample of the S-S-Eslicarbazepine was taken and given to mass spectrometry.

28.08.2018
The MS-Data was examined.
MS-Data suggested the reaction was not carried out as the original reactants were reobtained.

29.08.2018
The reactants were dried with a rotary evaporator for further use, but Thio-Eslicarbazepine was destroyed due to its instability in water.

30.08.2018
Destroying of the azide-donor.

31.08.2018
Cleaning