B013 Stability testing on senna and valerian dry extracts

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Quality criteria for the marketing authorisation of herbal medicinal products are permanently increasing. This includes the proof of sufficient stability of the respective plant material or its galenic formulation.

The presented work investigates the changes in quality of different plant extracts under the influence of environmental factors, such as increased temperature and humidity.

The materials included in this study were different methanolic and ethanolic dry extracts, according to the actual pharmacopoea and the corresponding herbal drug of valerian root, sennae pods and sennae leaves. The design of the stability testing was based on the ICH guidelines for stability testing of new drug substances and products (1), with the appropriate storage conditions of 25°C/60% RH, 30°C/60% RH and 40°C/75% RH.

TLC and HPLC fingerprint methods were utilized for investigating the changes in the chemical composition of extracts and herbal drugs. Due to the low content of some critical substances, SPE was used to separate and concentrate the found substances. The identification of the obtained products was confirmed by UV-spectra (HPLC-DAD) and mass spectrometry (LC-MS).

Distinct qualitative and quantitative changes in the fingerprint chromatograms were recognized for nearly all extracts examined under storage conditions of 40°C/ 75% RH. Depending on storage conditions, packaging form and additives, changes in powder conditions and hygroscopicity were recognized. Modifications of the physical parameters correlate with the changes in the fingerprint chromatograms of the extracts and the respective herbal drugs.

References: 1. Committee for proprietary medicinal products, CPMP/QWP/556/96, Note for guidance on stability testing of existing active substances and related finished products, 10/ 98.

B014 Quality control of *Eleutherococcus senticosus* roots: validation of an HPLC-method

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An HPLC method for the determination of eleutherosides B and E was developed based on published methods (1-3) and optimised. The extraction procedure, the extraction solvent and time and need for repeating the extraction till exhaustion were investigated. In the final method the powdered drug is heated on a water bath (60°C) for 30 min in 50% methanol. After cooling down, the solution is filtered into a round-bottomed flask. The residue is treated in the same way for a second and third time. The resulting filtrate is evaporated under reduced pressure until about 10 ml is left in the flask. This residue is quantitatively transferred into a volumetric flask and further diluted. A reversed phase HPLC system was used to evaluate the samples: column: RP-18; mobile phase: a gradient going in several stages from 90% phosphoric acid, water (0.5: 99.5) to 90% acetonitrile; detector: 220 nm.

Because eleutherosides B and E are not commonly commercially available ferulic acid was chosen as external standard. The correction factors for the response of ferulic acid against both eleutherosides were determined and validated (linearity and precision).

The method was fully validated, i.e. the linearity, the precision (repeatablility and intermediate precision on different days and at different concentration levels) and the accuracy (recovery) of the method were investigated and statistically evaluated.

This method, published in Pharmeuropa (4) is currently investigated by the European Pharmacopoeia Commission and open for remarks.

Acknowledgements: National Fund for Scientific Research – Flanders.

References: 1. Wagner, H. et al. (1982) Planta Med. 44: 193-8. **2.** Bladt, S et al. (1990) Dtsch. Apoth. Ztg. 130: 1499-508. **3.** Yat, P. et al. (1998) Phytochem. Anal. 9: 291-295. **4.** Eleutherococcus, PA/PH/Exp.13A/T (01)52ANP (2002) Pharmeuropa 14 (1): 104-5.