W07 Method validation for stability testing of *Boswellia* species and its preparations by HPTLC

A.J. Koch
Frohme – Apotheke, Frohmestr. 14, D – 22457 Hamburg, Germany

The exudates of Boswellic trees (Olibanum and Salai Guggul) belong to the group of gum resins. At the present time these gum resins are important commercial sources for pharmaceutical formulations. The active principles are the tirucallic acids (TA) and the boswellic acids (BA). They belong to the group of tetra- and pentacyclic triterpenes.
As there exists no actual monograph of Olibanum, according to the latests developments in science, the pharmaceutical companies have an interest in analytical methods for stability testing.
Gum resins belong to sticky materials and are not easy to handle. We propose HPTLC methods for the determination of the ketoBAs without derivatization by UV_{254} light and of BA's and TA's after derivatization. HPTLC - Fingerprints clearly differentiate one species from the other by the marker compounds. Multiwavelengthscans offer the possibilty to observe alterations, caused by the chromatographic process or by the manufacturing method, whereas 2- D –TLC in one single solvent system is suitable to attach the related compounds (of alteration) to each other.

W08 Qualification of TLC-systems for stability testing of *Vitex agnus-castus*-extracts

B. Meier
Zeller AG, Hebal Medicinal Products, CH.8590 Romanshorn. Switzerland.

TLC is one of the main issues to test stability of herbal medicinal products (HMP's) in future. The complex composition of HMP's can be documented with TLC. Newest guide-lines ask for tests, who characterise compounds of high, medium and low polarity of HMP's on several levels. This is a regulation more which overcomes the strategies of Ph Eur, which identifies herbal drugs with one specific TLC. Probably, a lot of additional TLC's will have to be developed in future.
Concerning stability testing, a main problem is the system qualification. Forced degradation tests are proposed to qualify TLC. The clinically approved (1) ethanolic dry extract of *Vitex agnus-castus* (Ze440) has been exposed to day-light, heat (50°C), hydrochloric acid, ammonium and humidity. Furthermore the extract has been dissolved in methanol and treated with light, 3% H_2O_2, 0,1 N trifluoro-acetic acid (TFA), 0.1 m dimethylamine (DMA) and heat (50°C/30 minutes). Iridoids disappeared completely under hydrochloric acid atmosphere. On the diterpene-TLC (2) rotundifuran disappeared under hydrochloric acid atmosphere, H_2O_2 and light. Linoleic acid was not detectable anymore after exposure to hydrochloric acid atmosphere and reduced under TFA und DMA. Hydrochloric acid atmosphere destroyed two further diterpenes, vitexilactone and 6β,7β-diacetoxy-13-hydroxy-labda-8,14-diene, meanwhile casticin was quite stable: Weaker peaks but not a complete decomposition have been observed under hydrochloric acid atmosphere and H_2O_2. C-glycosidated flavonoids, especially isoorientin, remained unchanged under all conditions, but chlorogenic acid was influenced into of the extract as well as a pure compound by hydrochloric acid atmosphere. The experimental work with forced degradation proved that chemical instabilities can be analysed by TLC and qualify the systems. Furthermore the results showed a relatively high robustness of the extract against physico-chemical influences.

Acknowledgements: The work was done in co-operation with CAMAG AG, Muttenz, and University of Basle (E. Reich, F.Wahli, W.Schaffner)

References: 1. Schellenberg R. (2001 British Medical Journal 322, 134-137. **2.** Dold U. (2000) HPTLC-Screening von Vitex agnus-castus-Früchten aus verschiedenen Herkünften. Diploma work, University of Basle.