Introduction

Herbal Dietary Supplements (HDS) are more popular now than ever before. The commercial success of botanical ingredients is largely due to a growing interest for products supporting a healthy lifestyle. HDS are widely marketed with various health claims which are often not compliant with regulations in force. Very popular are natural food supplements intended for weight-loss programs. However, such phytosupplements are easily available to consumers through several distribution channels: OTC in pharmacies, in supermarkets, herbalist’s shops or via the Internet.

With a very large and accessible market, the problem of adulteration of HDS with undeclared drugs is of growing importance, because HDS by definition have not been through the rigorous testing process which establishes the safety of a drug. The case of sibutramine (Fig. 1) is timely and of health concern (Fig. 2). This molecule was an approved drug used as an appetite suppressant. However in 2010, this drug was withdrawn from European [1] and US [2] markets because of its unacceptable risk over benefit ratio.

In this study, an HPTLC method was developed for the screening and determination of sibutramine in HDS purchased through the Internet.

Analysis

Extraction

1 capsule (about 300mg)
10ml of methanol
10’ ultra-sonication
10’ centrifugation

Identification

5ul sprayed on HPTLC Si 60 F254 (Merck)
with the Automatic TLC Sampler 4 (CAMAG)

Quantification

Range of medical dosage: 10 or 15 mg per day

This method has been compared with an in-house HPLC-ESI-MS/MS method (column : synergi polar 150mm x 2mm x 4um (Phenomenex), selected transitions (m/z) : 280.2→139.0 and 280.2→125.0, mobile phase : ammonium formate 5mM, pH4-4, methanol).

In this study, 39 weight-loss supplements were screened among which 17 were found to be adulterated with sibutramine. Amounts of drug detected ranged between 3 and 35 mg per capsule.

Quantities calculated (Fig. 8) with the HPTLC method are 14% higher in average in comparison to the HPLC-MS method. However, 10 samples have very similar results with both methods.

Conclusion and perspectives

The present study illustrates the alarming situation that almost half of the weight-loss supplements sold through the Internet were adulterated with the synthetic drug sibutramine. Undeclared pharmaceuticals are a serious problem of public health concern. This particularly since sibutramine became an unauthorized molecule in Europe, because it increases the risk of cardiovascular events.

This creates an urgent need for rapid screening methods that can detect pharmaceuticals in dietary supplements. Thus, this study demonstrates the usefulness of HPTLC to handled this issue.

In a near future, this type of methods need to be extended to other drugs (e.g. : rimonabant, orlistat, amphetamine derivatives).

Bibliography