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Characterization of Allegedly Musk-Containing Medicinal Products in Taiwan*

ABSTRACT: As a highly valued ingredient of Chinese medicinal remedies, musk is used as a detoxification agent and for treating fever, inflammation and swelling, and pain. Muscone (3-methylcyclopentadecanone-1), an odoriferous secretion from the ventral glands of male musk deer, is believed to be the active ingredient. A small amount of muscopyridine is also found in the secretion from the ventral glands of male musk deer. Common counterfeit ingredients are musk xylene, musk ambrette, musk ketone, and diphenhydramine. An extraction/GC-MS protocol/data evaluation scheme was developed and applied to study allegedly musk-containing Musk-Tiger Bone Plaster preparations and musk pods (or grains) from Chinese medicine stores and an airport customs. The content of muscone in a specific sample was estimated based on the percentage of the amount recovered from the first extraction. No muscone or counterfeit ingredients were found in all musk pod (or grain) samples from the customs and in the majority of Musk-Tiger Bone Plaster preparations, while muscone (alone or with counterfeit ingredients) was found in most of the musk pod (or grain) collected from Chinese medicine stores.

KEYWORDS: forensic science, muscone, musk deer, endangered species, illegal trade

Similar to bear bile (1), musk is a highly valued ingredient of Chinese medicinal remedies. It is reportedly an effective detoxification agent and can be used to reduce pain, inflammation, and swelling and to treat seizures (2–4). It is also used for some specialty perfumes and is among the most valuable animal products in the world at $45,000/kg (5).

Muscone, reportedly the active ingredient of musk (6), is a secretion of the preputial gland located under the abdomen near the pubic of the male musk deer (genus *Moschus*), of which at least four species have been identified: *M. moschiferus*, *M. berezovskii*, *M. chrysogaster*, and *M. fuscus*. Because of excessive hunting and destruction of forest habitat, the musk deer population has declined dramatically. Some populations now are included in Appendix I of CITES (Convention on International Trade in Endangered Species of Wild Fauna and Flora), while others are listed in CITES Appendix II. This means these deer and their derivatives are banned from (Appendix I) or require permits for (Appendix II) international trade.

Along with our earlier report on bear bile (1), this study was funded by and conducted as part of the Taiwanese Council of Agriculture's initiatives to conform with CITES' regulations and to understand the counterfeit status of musk-containing products in the market. We have conducted a literature review to better understand the ingredients of authentic and counterfeit musk products, collected representative samples, and developed an analytical and data analysis scheme suitable for this study. A consecutive extraction and data evaluation scheme was developed to determine the recovery efficiency of the adapted sample preparation procedure. This procedure was then used in conjunction with gas chromatography-mass spectrometry (GC-MS) protocols for the analysis of representative (allegedly) musk-containing products, and the findings are hereby reported.

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Methods and Materials

Specimens and Reagents

Specimens used in this study included 77 Musk-Tiger Bone Plaster preparations (Fig. 1) and 32 musk pods (or grains) (Fig. 2) collected from various Chinese medicine stores located in different parts of Taiwan and 17 allegedly musk glands obtained from the customs of an international airport. (Gland specimens are referred to as pods, while powder or crystalline materials allegedly resulting from the secretions of the gland are referred to as grains.)

All solvents and reagents were HPLC grade and were purchased from J. T. Baker, Inc. (Phillipsburg, NJ). A muscone standard was purchased from BIOMOL Research Lab (Plymouth Meeting, PA). Imipramine was obtained from Radian, now Cerilliant (Austin, TX).

Sample Preparation

Typically, 20 mg of musk grain or 200 mg of Musk-Tiger Bone Plaster were weighed and placed in a 10 × 75 mm glass test tube. Two mL of ethyl acetate were added. Extraction was carried out by placing the test tube in an ultrasonic system for 15 min, followed by centrifugation for 5 min. One mL of the supernatant was transferred to a 1.8-mL autosampler vial for GC-MS analysis.

Imipramine was used as the internal standard for the quantitation of muscone and muscopyridine. Specifically, 50 μL of a 100 μg/mL standard imipramine solution (in methanol), were added to the test tube before the extraction procedure proceeded. The concentration of the internal standard is equivalent to 5 μg/mL in the test sample.

Recovery of the extraction protocol was evaluated as follows. Following the first extraction, the residual solvent/extract was removed. Another 2-mL aliquot of ethyl acetate was added, and the