1. Introduction

Veterinary drugs are used for therapeutic and growth promotion purposes for animals or fishes. To provide assurance that food from animals is safe in regard to veterinary medicine residues, national authorities have established Maximum Residue Limits (MRL) for certain drugs in target tissues and animal species. Veterinary drug analysis commonly uses liquid chromatography coupled to mass spectrometer which is fast, highly sensitive and highly selective. This work describes the validation of high-throughput LC/MS/MS system utilizing fast and high precision total workflow was investigated with QuEChERS method combined with solid-phase extraction cartridges to enhance purification efficiency.

2. Methods and Pretreatment

Chicken, pork and beef were selected for recovery tests of veterinary drugs. Evaluation of analytical system and recovery test used 129 veterinary drugs spiked in meat (1.25 ppb in vial). Solid phase extraction Technique with QuEChERS method (20 mg of L400 fully automated solid phase extraction system (ST-440, AISII SCIENCE), LC and MS conditions are shown in Table1. ODS column and Biphenyl column were used to evaluate the peak shape comparison of ODS column and Biphenyl column are shown in Fig4. Biphenyl column achieved good peak separation for alpha-tretonine and beta-trenbolone using ODS column and biphenyl column within 18 minutes. MRM chromatogram of alpha- and beta-trenbolone, and sulfisoxazole and sulfamethazine using ODS column and biphenyl column are shown in Fig3. Biphenyl column achieved good peak separation for alpha-trenbolone and beta-trenbolone. Conversely, ODS column sufficiently separated sulfisoxazole and sulfamethazine. Adequate selection of column enables to establish accurate quantitative analytical system.

3. Results

Comparison of ODS column and Biphenyl column

In the Fig5 below, both ODS column and Biphenyl column measured all of 129 veterinary drugs from standard sample (10 ppb) within 18 minutes. MRM chromatogram of alpha- and beta-trenbolone, and sulfisoxazole and sulfamethazine are compared in Fig5. Biphenyl column achieved good peak separation for alpha-trenbolone and beta-trenbolone. Conversely, ODS column sufficiently separated sulfisoxazole and sulfamethazine. Adequate selection of column enables to establish accurate quantitative analytical system.

Recoveries of Veterinary Drugs in Chicken, Pork and Beef

Purified extract from chicken, pork and beef were assayed using LC/MS/MS using ODS column. The peak area of standard and post-spike sample were compared for matrix effects. The peak area of pre-spiked sample and post-spike sample were compared for recovery rates. The concentration of standard, pre-spiked and post-spike sample were diluted at the concentration of 1.25 ppb. 94 drugs were obtained from each sample. The results indicated that 82% of the compounds in chicken, 85% of the compounds in pork and 84% of the compounds in beef were recovered from 70 to 120% (n=3). Table2 shows the typical result of matrix effects and recovery test from each samples. Stable and good recoveries were achieved with fully automated STQ method.

Table1. ODS column and Biphenyl column were used to evaluate the peak shape comparison of ODS column and Biphenyl column in meat (1.25 ppb in vial). Solid phase extraction Technique with QuEChERS method (20 mg of L400 fully automated solid phase extraction system (ST-440, AISII SCIENCE), LC and MS conditions are shown in Table1. ODS column and Biphenyl column were used to evaluate the peak shape comparison of ODS column and Biphenyl column are shown in Fig4. Biphenyl column achieved good peak separation for alpha-tretonine and beta-trenbolone using ODS column and biphenyl column within 18 minutes. MRM chromatogram of alpha- and beta-trenbolone, and sulfisoxazole and sulfamethazine are compared in Fig3. Biphenyl column achieved good peak separation for alpha-trenbolone and beta-trenbolone. Conversely, ODS column sufficiently separated sulfisoxazole and sulfamethazine. Adequate selection of column enables to establish accurate quantitative analytical system.

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Table2. Typical result of matrix effects and recovery test (%)

Table3. Effect of matrix on recovery rate (%)

Table4. Effect of matrix on recovery rate (%)