



## Determination of 19 Quinolone Residues in Honey

### 1. Scope of application

Used for determination of 19 quinolone residues in honey

### 2. Sample preparation

#### 2.1 Internal standard stock solution of deuterated Norfloxacin (NOR-D5):

Dissolve proper amount of NOR-D5 standard in methanol to obtain internal standard stock solution of 100 µg/mL. Dilute proper amount of the internal standard stock solution with methanol to obtain working solution at the concentration of 1 µg/mL and store at 4°C.

#### 2.2 Preparation loading sample

Weigh 5g of sample and add to a 50mL of centrifuge tube with stopper. Add 50µL of 1µg/mL internal standard solution and 5mL of 0.1mol/L sodium hydroxide solution. Vortex mix to dissolve the honey thoroughly.

### 3. Sample purification

Chrompure PAX 60 mg/3mL (LBSPAX603)

Condition: 5mL of methanol followed by 3mL of water

Load: add loading sample

Wash: 3mL methanol/3mL water

Elute: 3mL of methanol containing 5% formic acid

Reconstitute: Collect the eluate and evaporate to dryness by nitrogen at 40°C. Dilute to 1.0mL with 20% methanol in water. Filter through 0.45µm membrane to sample vial for LC-MS analysis.

## Determination of Nitrofurantoin Residues in Foods

### 1. Scope of application

Used for determination of nitrofurantoin residues in foods

### 2. Sample preparation

(1) Treatment on milk powder and milk

add 15mL of solution mixed by trichloroacetic acid and water (2 and 1, 5mol) in milk powder of 1g (milk of 5g); append the interior label and mixed label; water bath and hydrolyzation at 37.5°C for 5h; centrifuge at 4000 rpm for 5min and then get the supernate for usage. activate the PEP cartridge with 5mL of methanol and 5mL of water; pass the treated supernate in SPE cartridge and wash the cartridge with 5mL of



trichloroacetic acid; collect the solution into another test tube; derive the derivating agent of 100 $\mu$ l (dissolve 20mg 2-nitrobenzaldehyde in dimethylsulfoxide of 1mL) at 37.5 $^{\circ}$ C for 16h in the water bath( overnight)

(2) Treatment on the samples of pork, beef, chicken, pork liver, aquatic product and honey

add 15mL mixed solution of methanol and water in 2g of pork, beef, chicken, pork liver and aquatic product respectively (honey of 5g) and vortex; centrifuge at 4000 rpm for 5min and add the interior label and mixed label into the supernate. add derivating agent(dissolve 20mg 2-nitrobenzaldehyde in dimethylsulfoxide of 1mL)of 100 $\mu$ L into the supernate; derivate the solution in the water bath of 37.5 $^{\circ}$ C for 16h (overnight).

### 3. Sample purification

Chrompure PLS 60 mg/3mL (LBSPLS603)

**Condition:** 5mL methanol/5mL water

**Load:** add the buffer solution of dipotassium phosphate 5mL in the derivative solution; adjust the pH to 7.4 with 1mol/L of sodium hydroxide solution; centrifuge at 4000r/min for 10min; keep the supernate(If the sample contain much fat, the supernate should be added n-haxane of 5mL; absorb and remove n-haxane by vibration for 2min and centrifuging at 4000rpm for 10min) going through the PLS column with the flow rate less than 2mL/min

**Wash:** 10mL water

**Elute:** 5mL Ethylacetate

**Reconstitute:** dry the eluate with nitrogen at 40 $^{\circ}$ C; dissolve the solution and fix the volume to 1.0mL with the sample of constant volume solution; filter through the filter membrane of 0.2 $\mu$ m. Then analysis with LC-MS.

## Determination of 8 Uretic Residues in the Animal Urine

### 1. Scope of application

Used for determination of nitrofurans residues in foods

### 2. Sample preparation

Transfer 2 mL of sample accurately to a 50-mL centrifuge tube. Adjust pH with 5 mol/L hydrogen chloride solution to 3.5 $\pm$ 0.5. Add 1 mL of 5% lead acetate solution and 5 mL of water-saturated acetic ether. Vortex mix and vibrate on shaker for 10 min. Centrifugate at 5000 rpm for 5 min and transfer the supernatant to another 50-mL centrifuge tube. Add 5 mL of water-saturated acetic ether to the aqueous underlayer. Vortex mix and vibrate on shaker for 10 min. Centrifugate at 5000 rpm for 5 min and combine the supernatants. Dry the solution under nitrogen at 50 $^{\circ}$ C. Dissolve the residue in 3 mL of acetonitrile-2% ammonia (10:90, V/V) for later use.

### 3. Sample purification



Chrompure PAX 60 mg/3mL (LBSPAX603)

**Condition:** 3mL methanol/3mL 2% ammonia.

**Load:** add sample

**Wash:** 3 mL of 2% ammonia/3mL methanol

**Elute:** 3 mL of methanol containing 5% formic acid

**Reconstitute:** Dry the eluate under nitrogen. Dissolve the residue in 1mL of acetonitrile-0.3% ammonia(10:90, V/V). Vortex mix and filter the solution through 0.22  $\mu$ m membrane for LC-MS/MS analysis.

## Determination of Sulfonamides in Animal Tissues

### 1. Scope of application

Used for determination of determination of sulfonamides in animal tissues

### 2. Sample preparation

Add 5g tissues sample into 50ml centrifuge tube, and add 25mL ethyl acetate, 5g anhydrous sodium sulfate. Homogenize for 2minute, centrifugate at 4000r/min for 2min, transfer the ethyl acetate layer. Extract the sample paste again using 25mL ethyl acetate, homogenize for 2minute, centrifugate at 4000r/min for 2min, transfer the ethyl acetate layer. Combine the ethyl acetate layer. Evaporate the solution to dryness under rotovap. Reconstitute the residue in 2ml 1%ethyl acetate, 1mL methanol and 3mL n-hexane, Vortex 1min, evaporate the solution to dryness under rotovap. Reconstitute the residue in 2ml 1%ethyl acetate, 1mL methanol and 3mL n-hexane, Vortex 1min and centrifuge for 2 min at 4,000 rpm. Discard the n-hexane layer, then add 6ml n-hexane, repeat the operation. Finally add 6mL lower layer. Then get the layer for usage.

### 3. Sample purification

Chrompure PLS 60 mg / 3mL (LBSPLS603)

**Condition:** 3 mL MeOH / 3 mL H<sub>2</sub>O

**Load:** Add sample

**Wash 1:** 3 mL H<sub>2</sub>O

**Wash 2:** 3 mL MeOH:H<sub>2</sub>O = 5:95

**Elute:** 5 mL MeOH

**Reconstitute:** Evaporate at 30 °C by N<sub>2</sub>, reconstitute to 1 mL with mobile phase and filter the solution through 0.22  $\mu$ m membrane for LC-MS/MS analysis.



## Determination of Sulfonamides in Honey

### 1. Scope of application

Used for determination of determination of sulfonamides in honey

### 2. Sample preparation

Weigh 5g of sample in a 50mL centrifuge tube. Add 15mL of water and vibrate on shaker for 10 min. Use 40mL ethyl acetate extracted for two times. Collecting the ethyl acetate layer. Vacuum evaporate the ethyl acetate layer to near dry at 30°C, Dissolve the residue in 10mL of 2% phosphate, filter the solution through 0.22 µm membrane for usage.

Chrompure PCX 200mg/6mL (LBSPCX2006)

Condition: 6mL MeOH/6mL H<sub>2</sub>O

Load: Add sample

Wash 1: 6mL H<sub>2</sub>O

Wash 2: 6mL MeOH

Elute: 6mL 5% NH<sub>4</sub>OH in MeOH

Reconstitute: Vacuum evaporation at 30°C, reconstitute to 1mL with mobile phase, Then analysis with HPLC.

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