Original: Japanese
Provisional translation

Analytical Method for Quinclorac (Animal Products)

1. **Analyte**
   Quinclorac
2. **Applicable food**
   Animal products
3. **Instrument**
   Liquid chromatograph-tandem mass spectrometer (LC-MS/MS)
4. **Reagents**
   Use reagents listed in Section 3 of the General Rules, except the following.
   Reference standard of quinclorac: Contains not less than 98% of quinclorac.
5. **Procedure**
   Add 100 mL of acetone/hydrochloric acid (99:1, v/v) to 10.0 g of sample, homogenize, and filter with suction. Add 50 mL of acetone/hydrochloric acid (99:1, v/v) to the residue on the filter paper, homogenize, and filter as described above. Combine the resulting filtrates, and add acetone to make exactly 200 mL. Take a 4 mL aliquot of the solution accurately, and add 40 mL of 10 w/v% sodium chloride solution containing 2 w/v% sodium hydrogen carbonate. Add 40 mL of ethyl acetate, shake, remove the ethyl acetate layer, and repeat this step one more time. Add 1 mL of hydrochloric acid to the aqueous layer, and extract with shaking twice with 40 mL and 20 mL of ethyl acetate. Combine the extracts, dehydrate with anhydrous sodium sulfate, and filter out the anhydrous sodium sulfate. Concentrate the filtrate at below 40°C, and remove the solvent. Dissolve the residue in methanol to make exactly 4 mL, and use this solution as the test solution.
6. **Calibration curve**
   Prepare quinclorac standard solutions (methanol) of several concentrations. Inject each standard solution to LC-MS/MS, and make calibration curves by peak-height or peak-area method. When the test solution is prepared following the above procedure, the sample containing 0.01 mg/kg of each analyte gives the test solution of 0.0005 mg/L in concentration.
7. **Quantification**
   Inject the test solution to LC-MS/MS, and calculate the concentration of quinclorac from the calibration curve made in 6.
8. **Confirmation**
   Confirm using LC-MS/MS.
9. **Measurement conditions**
   (Example)
   Column: Octadecylsilanized silica gel (2.1 mm in inside diameter, 150 mm in length, 3 μm in particle diameter)
   Column temperature: 40ºC

Mobile phase: Linear gradient from 5 mmol/L ammonium acetate/5 mmol/L ammonium acetate-methanol (9:1, v/v) to (1:19, v/v) in 20 minutes.
Ionization mode: ESI (+)
Major monitoring ion (*m/z*): Precursor 242, product 196, 161
Injection volume: 2 μL
Expected retention time: 11 minutes

10. Limit of quantification

0.01 mg/kg

11. Explanatory note

1) Outline of analytical method

The method consists of extraction of quinclorac from sample with acetone under acidic conditions of hydrochloric acid, wash with ethyl acetate under basic conditions, transferring into ethyl acetate under acidic conditions, and quantification and confirmation using LC-MS/MS.

2) Notes

i) When the analytical method for quinclorac using LC-MS/MS was developed, the following monitoring ions were used:
for quantitative ions *(m/z)*: precursor ion 242, product ion 161
for qualitative ions (*m/z*): precursor ion 242, product ion 196

ii) It foams vigorously when hydrochloric acid is added to the aqueous layer, therefore shake after the foam settles.

iii) Food items used to develop the analytical method: Cattle muscle, cattle fat, cattle liver, milk, egg

12. References

None

13. Type

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