A method of liquid chromatography coupled with tandem mass spectrometry (LC-MS/MS) was developed for the determination of 3,5-dinitrosalicylic acid hydrazine (DNSH), which was the metabolite of nifursol antibiotic in animal origin food. The samples were hydrolyzed with 0.1 mol/L HCl, and derivatised with 2nitrobenzaldehyde at 37 °C for 16 h. The derivative solutions were adjusted to pH 7.0—7.5, and extracted by ethyl acetate. The analyte was detected by tandem mass spectrometry with electrospray ionization source by MRM mode. There is good linear correlation between the peak areas and concentrations of DNSH(the calibration coefficient is 0.999 5), the dynamic linear range is 0.5—10 μ g/kg. The limit of detection (S/N=3) is 0.5 μ g/kg. The recoveries of DNSH at four spiked levels of 0.5, 1.0, 2.0, 4.0 μ g/kg range from 63.4% to 109.5% (*n*=6) and the RSDs are between 2.0% and 11.9% (*n*=6). It is proved to be fast and effective for simultaneously qualitative and quantitative inspection of the metabolite of nifursol antibiotic in animal origin food.

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Determination of the Metabolite of Nifursol in Animal Origin Food by HPLC-MS/MS		
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