

# Validasi analisis residu Pestisida Karbaril, Klorpirifos, dan Dimetoat dalam buah menggunakan metode QuEChERS dan LC-MS/MS = Validation of Pesticide residue analysis Carbaryl, Chlorpyrifos and Dimethoate in fruit using QuEChERS and LC-MS/MS method

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## Abstrak

Pestisida dipercaya dapat menurunkan populasi hama dengan cepat sehingga meluasnya hama dapat dicegah. Residu pestisida menimbulkan efek yang dalam jangka panjang dapat menyebabkan gangguan kesehatan berupa gangguan pada sistem syaraf serta metabolisme enzim. Tujuan penelitian ini adalah untuk menentukan kadar residu pestisida yang terdapat dalam buah apel, anggur, pir dan stroberi. Sebuah metode Liquid Chromatography-tandem Mass Spectrometry (LC-MS/MS) yang cepat dan sensitif, dalam mode ionisasi elektrospray positif, telah dikembangkan untuk penentuan multi-kelas pestisida. Ekstrak diperoleh dengan menggunakan asetonitril dengan teknik preparasi sampel berbasis QuEChERS (quick, easy, cheap, effective, rugged and safe). Untuk acuan uji digunakan 3 jenis bahan aktif pestisida dari 2 golongan organofosfat dan karbamat.

Dari metode validasi yang dilakukan, diperoleh nilai koefisien korelasi,  $R^2$  0,996 dengan rentang 0-1000 ppb dan 0-1 ppm. Rentang batas deteksi (LOD) dan batas kuantisasi (LOQ) dari ketiga senyawa berkisar dari konsentrasi 0,01 ppm - 0,215 ppm. Persen perolehan kembali berkisar antara 38%-44% dengan standar deviasi relatif <16%. Persen perolehan kembali yang rendah dikarenakan komponen matriks yang terdapat dalam sampel dan juga nilai LOD yang rendah sehingga sensitivitas alat berkurang.

Berdasarkan hasil analisis, pestisida dimetoat tidak terdapat dalam sampel buah anggur, stroberi, dan pir yang telah diuji. Sedangkan, residu pestisida karbaril dan klorpirifos ditemukan dalam buah-buahan tersebut dengan intensitas yang sangat rendah sehingga tidak terdeteksi oleh alat karena berada dibawah batas deteksi. Sehingga disimpulkan bahwa buah-buahan tersebut masih aman untuk dikonsumsi karena berada dibawah Batas Maksimum Residu yang ditetapkan oleh Badan Standardisasi Nasional dalam SNI 7313:2008.

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Pesticides are believed to reduce the population of pests quickly so the widespread of pests can be prevented. The effects of pesticide residues in the long term can lead to health problems such as interference with the nervous system and metabolic enzymes. The purpose of this research is to determine the pesticide residues found in apples, grapes, pears and strawberries. Samples taken at random, from traders and supermarkets in the area of Depok. A rapid and sensitive liquid chromatography-tandem mass spectrometry method, in electrospray ionization positive mode, has been developed for the determination of selected multi-class pesticides. Extracts were obtained using the acetonitrile- based QuEChERS (quick, easy, cheap, effective, rugged and safe) sample preparation technique. For the reference test used 3 types of active ingredients from two groups of pesticide which is organophosphates and carbamates.

Based on the validation methods, R<sup>2</sup> values obtained 0,996 with a range of 0-1000 ppb and 0-1 ppm. Limit of Detection (LOD) and Limit of Quantitation (LOQ) of the third compound concentrations ranged from 0,01 ppm - 0,215 ppm. Percent recoveries ranging from 38%-44% with relative standard deviations <16%. Percent recoveries was low due to matrix components present in the sample and a low LOD's value so that the instrument sensitivity is reduced. Data analysis samples were then interpreted, and the figures obtained were compared with standard Maximum Residue Limit pesticides listed in ISO 7313:2008.

Based on the analysis, pesticide dimethoate was not present in samples of grapes, strawberries, and pears that have been tested. However, karbaril and chlorpyrifos pesticide residues found in these fruits with a very low intensity so that was not detected by the instrument because it is below the limit of detection. Thus concluded that the fruit is still safe to eat because they are under Residue Limit set by Badan Standardisasi Nasional in SNI 7313:2008.