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VALIDATION OF THE METHOD FOR THE DETERMINATION OF DITHIOCARBAMATES IN FRUITS

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Residues of active substances, which were frequently found in fruits and vegetables of market producers belonged to the dithiocarbamate group pesticide. According to their use dithiocarbamates belong to fungicides in terms dithiocarbamic acid. They are a group of compounds that exist as strong complexes with various metal ions, often in a polymeric form. This makes them difficult to analyse directly because of their limited solubility in most organic solvents. It is important to have a reliable method which enables the quick and simple detection of this group for official monitoring. This work reports a simple, rapid and sensitive method for the assessment residues dithiocarbamates in fruits. Dithiocarbamates were determined indirectly by measuring the amount of carbon disulfide (CS_2) that is liberated by the chemical reaction. The fruit samples in undisturbed condition were heated with a solution of stannous (II) chloride and hydrochloric acid yielding carbon disulphide. Incurred carbon disulfide (CS_2) are determined "head-space" technique of gas-chromatography with mass detector. Linearity was verified by using the solutions of carbon disulphide in acetone. A linear dynamic range was obtained over a range of concentrations from 0.02 to 0.12 mg/kg for carbon disulfide with correlation coefficient $r > 0.995$. The accuracy of the method was acceptable since the average recoveries measured at four fortification levels were in the range of 83-103% ($n = 4$). The precision of the developed procedure expressed as the relative standard deviations (RSDs) were lower than 3.7% in all cases. Quantification was based on external standard calibration curves made with spiked blank-matrices.

Key words: apple, validation, dithiocarbamates, GC-MS

VALIDACIJA METODE ZA ODREĐIVANJE DITIOKARBAMATA U VOĆU

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Ostaci aktivnih supstanci koje su često nalaze u voću i povrću na tržištu proizvođača često pripadaju ditiokarbamatima. Ditiokarbamati su derivati ditiokarbaminske kiseline i ubrajaju se u fungicide. To je grupa jedinjenja koja se javlja u obliku kompleksa sa različitim jonima metala često u polimernoj formi. Sve su to razlozi zbog čega je ograničena njihova rastvorljivost u većini organskih rastvarača i samim tim ih je teže direktno analizirati. Važno je imati pouzdan metod koji omogućava brzu i jednostavnu detekciju ove grupe jedinjenja za njihovo dalje praćenje. Ovaj rad predstavlja jednostavan, brz i osetljiv metod za procenu ostataka ditiokarbamata u voću.

Ditiokarbamati su određeni indirektno merenjem količine ugljen-disulfida (CS_2) koji se oslobodio u hemijskoj reakciji. Uzorci voća u nenarušenom stanju su zagrevani sa rastvorom kalaj (II)-hlorida i hlorovodonične kiseline pri ćemu je nastao ugljen-disulfid. Nastali ugljen-disulfid je određen "head-space" tehnikom gasne hromatografije sa masenim-detektorom. Linearnost je potvrđena korišćenjem rastvora ugljen disulfida u acetonu. Linearni dinamički opseg je dobijen u opsegu koncentracija od 0,02 do 0,12 mg /kg za ugljen-disulfid sa koeficijentom korelacije $r > 0,995$. Tačnost metode je prihvatljiva izmeren je prosečan povrat uz standardni dodatak koji se kretao u opsegu od 83-103% ($n = 4$). Preciznost razvijene metode izražene kao relativna standardna devijacija (RSDs) iznosio je manje od 3,7% u svim slučajevima. Kvantifikacija je zasnovana na standardnoj kalibracionoj krivi sa spoljnim standardom koja predstavlja standardni dodatak na matriks.

Ključne reči: voće, validacija, ditiokarbamati, GC-MS