

# Method Validation for Simultaneous Quantitative Analysis of Acetaminophen and Dexamethasone in Jamu Pegal Linu Using SPE-HPLC Method

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## Abstract

### Backgrounds :

The issue of chemical adulterations in jamu in Indonesia happens every year. Chemical adulterations cases mostly were found in jamu pegal linu (Indonesian herbal medicine for treating muscle aches). The available standard methods for chemical adulterations analysis in jamu are only for their qualitative analysis.

### Aims :

This research was aimed to develop quantitative analysis method for chemical adulterations in jamu pegal linu, using solid phase extraction (SPE) and high-performance liquid chromatography (HPLC).

### Methods :

Sample preparation used SPE OASIS HLB with ammonia 2.5% in methanol as eluent. HPLC was carried out using the C-18 column as stationary phase, methanol: water as mobile phase, gradient elution type, UV detector at a wavelength of 254 nm and flow rate of 1 mL/minute. Results :

The linearity of standard curves for acetaminophen in the ranges between 10 – 100 ppm and dexamethasone in the ranges between 0.5 – 5 ppm were good. Specificity, precision, limit of detection and limit of quantification for acetaminophen and dexamethasone, meet the criteria. As for accuracy, only acetaminophen analysis fulfilled the requirement.

### Conclusion :

Method validation of acetaminophen and dexamethasone has been conducted. All of validation parameters were met the requirements except for accuracy parameters, only quantitative analysis of acetaminophen met the requirement

**Keywords:** Jamu pegal linu, HPLC, acetaminophen, dexamethasone, SPE

## INTRODUCTION

Jamu is Indonesian herbal medicines which have been used for generations. According to Indonesian Ministry of Health Regulation No. 007 in 2012, jamu should not contain chemical drugs including natural chemical isolate nor synthetic chemical compounds: ethanol should not exceed more than 1.0%. Chemicals adulteration cases are difficult to be eliminated. Based on Indonesian National Agency for Drugs and Foods Controls in 2001 – 2007, the trend of chemicals contamination in jamu led of jamu pegal linu. The two compounds that mostly found in jamu pegal linu were acetaminophen and dexamethasone. Until 2014, acetaminophen was the most frequently chemical compound to be found in jamu [1, 2, 3]. Quantitative analysis of chemical adulterations in jamu is needed for monitoring the amount of the added chemicals in jamu also for the study of its exposure level to jamu consumers. Hence, the society could get precise information of the chemicals adulteration contained in jamu.

Acetaminophen,  $C_8H_9NO_2$ , chemical name: 4-hydroxyacetanilide; p-hydroxy acetanilide; p-acetamidophenol; p-acetaminophenol; p-acetylamino phenol; N-acetyl-p-aminophenol [5].

Description: white crystalline powder, odorless, slightly bitter. Solubility: freely soluble in alcohol, soluble in boiling water and NaOH 1 N [9, 10].

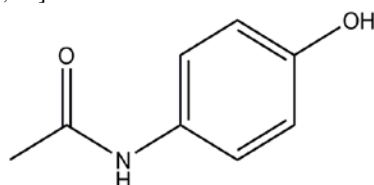


Fig.1: Molecular structure of acetaminophen

Dexamethasone,  $C_{22}H_{29}FO_5$ , description: white to almost white powder, odorless, crystalline powder, stable at room temperature, decompose at 250 °C. Solubility: Sparingly soluble in acetone, alcohol, dioxan, and methanol, slightly soluble in chloroform,

very slightly soluble in ether, practically insoluble in water [6, 10].

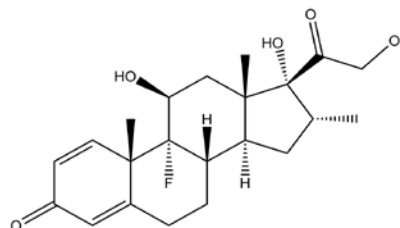


Fig.2: Molecular structure of dexamethasone

The available standard methods of analysis for monitoring chemical adulterations in jamu are thin layer chromatography (TLC) and TLC-densitometry [10]. These methods are only for identification of the chemicals in jamu. Currently, quantitative methods analysis developments for chemical adulterations in jamu are still limited. SPE was chosen for sample preparation considering its ability to separate analytes from complex matrices such as herbal products. HPLC was used to analyzing chemicals in jamu because this method has good accuracy for quantitative and qualitative chemicals analysis.

## MATERIALS AND METHODS

### Materials

The analytical reference of acetaminophen and dexamethasone are obtained from the pharmaceutical industry. All reagents for preparation of HPLC mobile phase were chromatographic grade and for the other procedures were analytical grades (Merck). SPE was performed using OASIS HLB 60 mg 3 mL (Waters).

### Crude Drugs

The crude drugs, i.e. Curcumae xanthorrhizae rhizome, Curcumae domesticae rhizome, and Zingiberis officinalis rhizome, were purchased from the local herbal store. The crude drugs were obtained as a powder.