METHODSNOW™
A CAS SOLUTION

JAICI
化学情報協会

CAS®
A DIVISION OF THE
AMERICAN CHEMICAL SOCIETY

分析手法の検索にお困りではないですか？

MethodsNow (メソッズナウ) とは

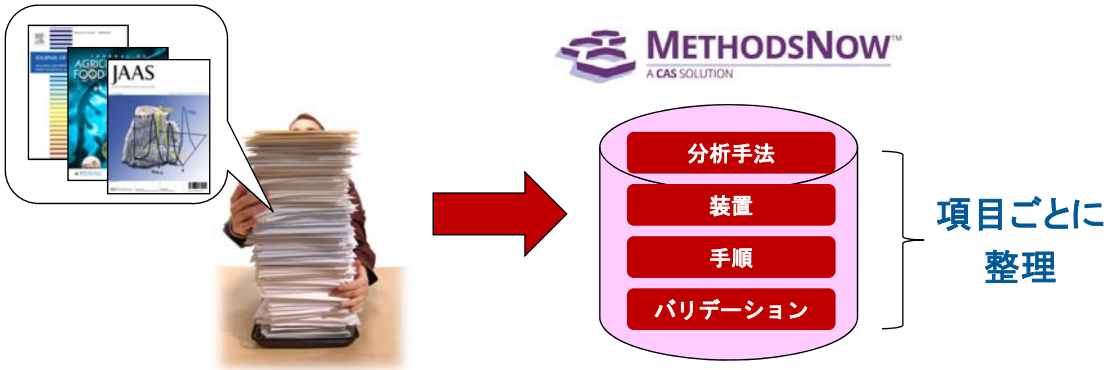
- 米 CAS (Chemical Abstracts Service) が新たに構築した分析手法に関する世界最大のデータベース
- 必要な情報を簡単に，素早く，正確に入手

JAICI 化学情報協会

CAS A DIVISION OF THE AMERICAN CHEMICAL SOCIETY

MethodsNow (メソッズナウ) とは

CAS が保有する文献コレクションから情報を抽出し，データベース化



原文献を読む手間を削減し，業務効率アップ！

JAICI 化学情報協会

CAS A DIVISION OF THE AMERICAN CHEMICAL SOCIETY

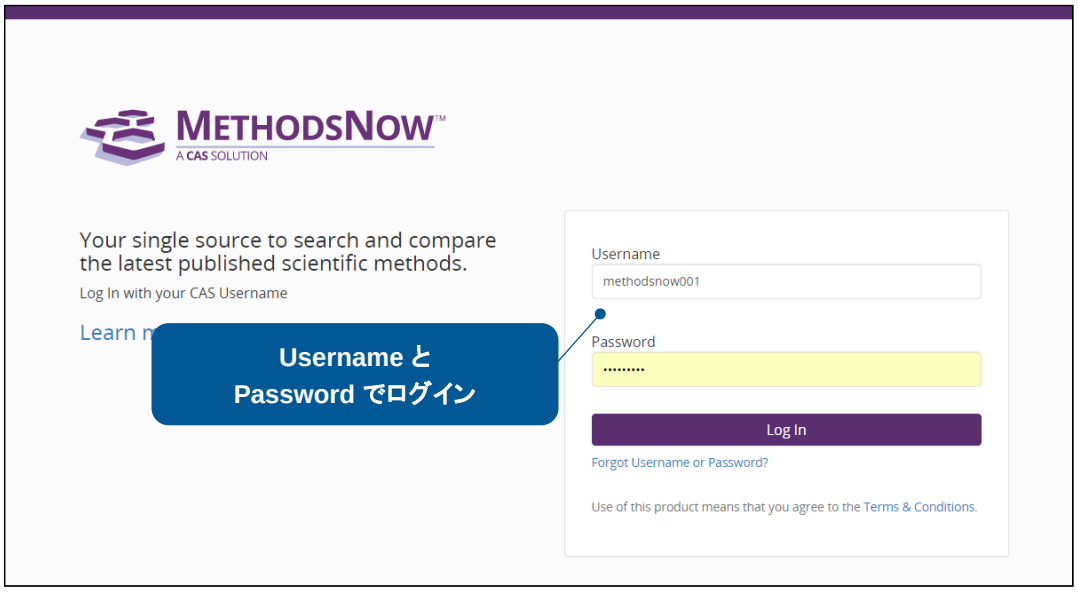
MethodsNow の利用シーン

- より簡単で効率的な分析手法を調べたい
- ある物質に関する分析手法を比較したい
- 所有している分析機器で可能な分析手法を確かめたい

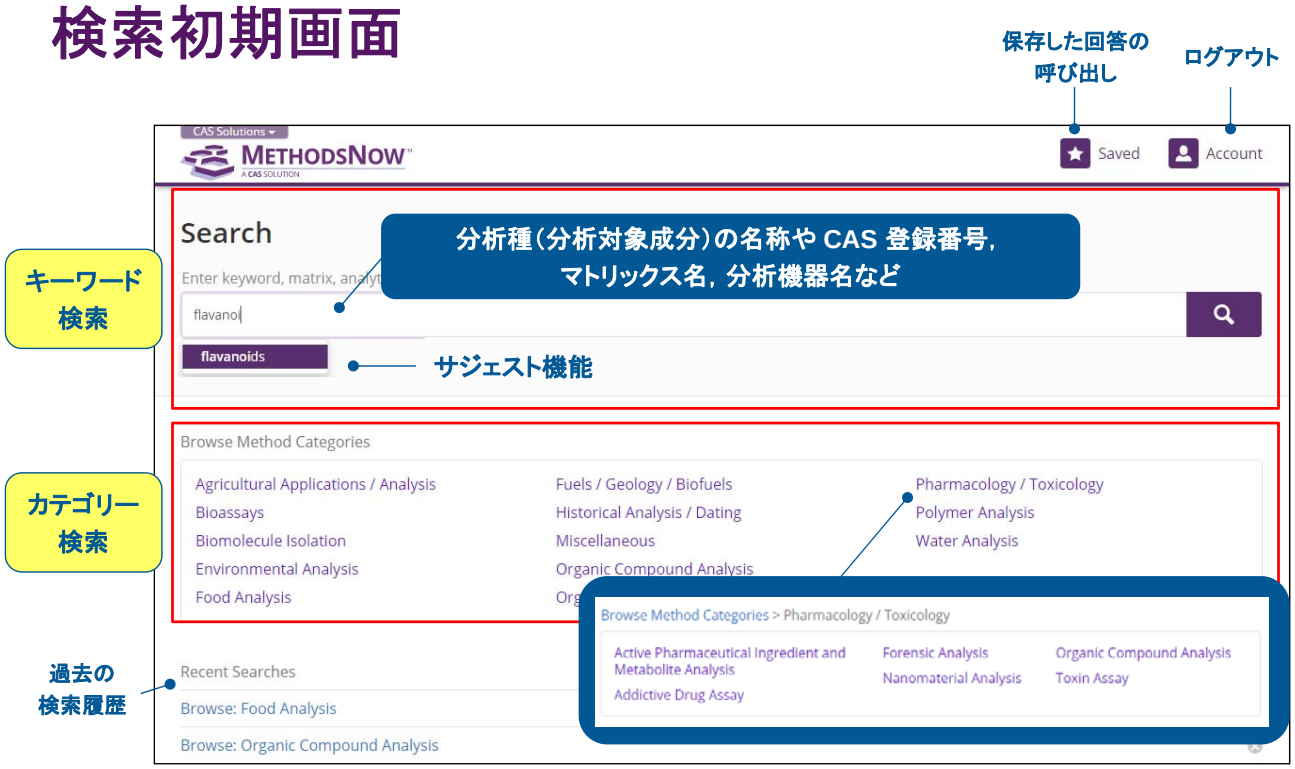
分析に関する詳細な情報を簡単に入手したい方に！

Analysis インターフェース

https://www.methodsnow.com/



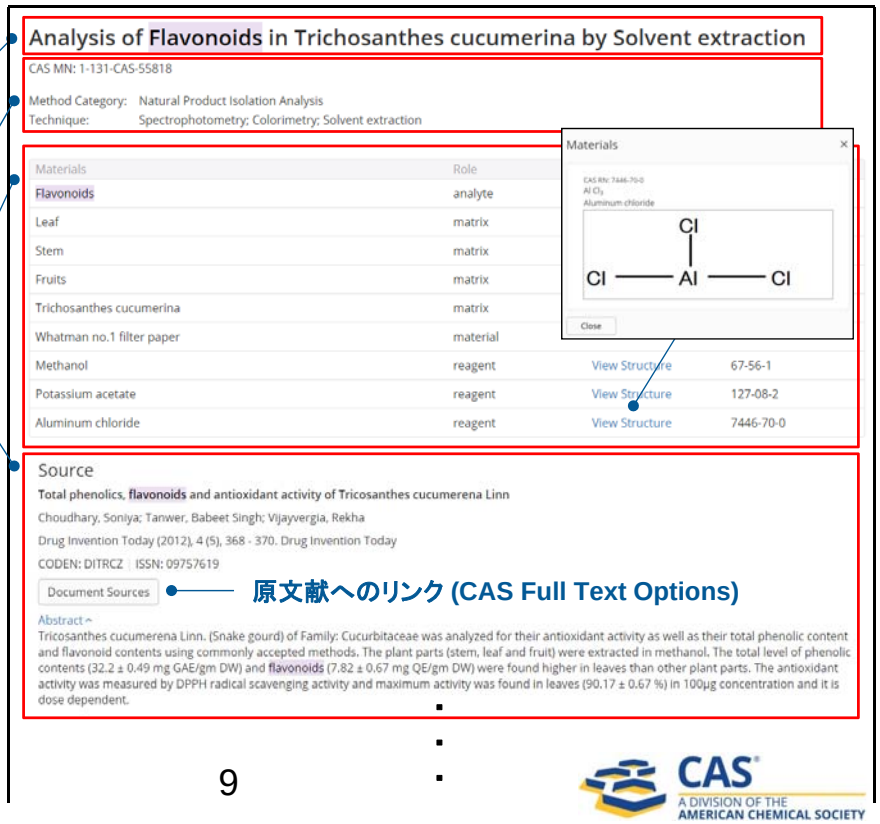
検索初期画面



結果一覧画面

結果詳細画面

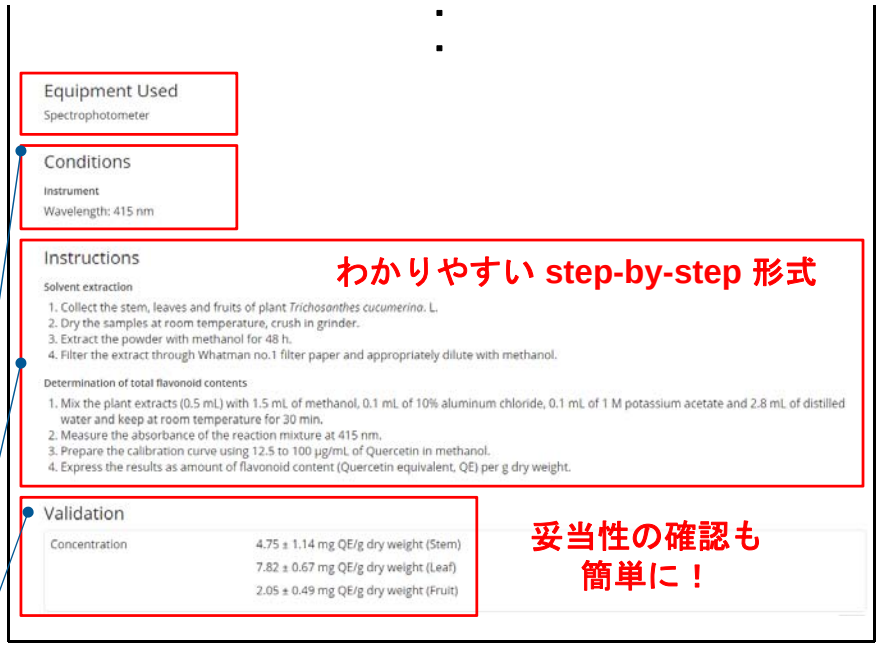
- タイトル
- CAS Method 番号
  分析カテゴリー
  分析手法名
- 物質情報
- 収録源
  （雑誌名，著者名，
  出典のタイトル，
  抄録など）
- 使用機器，分析条件
- 分析手順
- バリデーション



原文献へのリンク (CAS Full Text Options)

結果詳細画面

- タイトル
- CAS Method 番号
  分析カテゴリー
  分析手法名
- 物質情報
- 収録源
  （雑誌名，著者名，
  出典のタイトル，
  抄録など）
- 使用機器，分析条件
- 分析手順
- バリデーション



わかりやすい step-by-step 形式

妥当性の確認も
簡単に！

結果の比較機能

Compare (3/3)

最大 3 つまで比較可能！

	1	2	3
Title	Analysis of Flavonoids in Trichosanthes cucumerina by Solvent extraction	Analysis of Flavonoids in Salacia chinensis by Solvent extraction	Analysis of Flavonoids in Prunus persica by Fractionation
CAS Method Number	1-131-CAS-55818	1-131-CAS-119913	1-131-CAS-140982
Method Category	Natural Product Isolation Analysis	Natural Product Isolation Analysis	Natural Product Isolation Analysis
Technique	Spectrophotometry; Colorimetry; Solvent extraction	Spectrophotometry; Solvent extraction	Fractionation; Colorimetry; Solvent extraction
Analyte	Flavonoids	Flavonoids	Flavonoids
Matrix	Leaf; Stem; Fruits; Trichosanthes cucumerina	Fibrous materials; Fruits; Salacia chinensis	Fruits; Prunus persica
Other Materials	Meti Alun filter	Acetone; Potassium acetate; Aluminum chloride; Whatman No. 1 filter paper	Acetone; Sodium hydroxide; 1-Butanol; Aluminum trichloride hexahydrate; Sodium nitrite; Buchner View All
quipment Used	Spectrophotometer	UV-spectrophotometer, 190 double beam, Shimadzu, Japan	Spectrophotometer, Pharmaspec UV - 1700, Shimadzu, Kyoto, Japan
Conditions	**Instrument:** Wavelength: 415 nm	**Instrument:** Wavelength: 415 nm	**Instrument:** detection wavelength: 510 nm

分析手法

分析対象

マトリックス

使用機器や分析条件の比較

使用機器

分析条件

結果の比較機能

具体的な分析手順

	1	2	3
Method	**Solvent extraction** 1. Collect the stem, leaves and fruits of plant *Trichosanthes cucumerina*. L. 2. Dry the samples at room temperature, crush in grinder. 3. Extract the powder with methanol for 48 h. 4. Filter the extract through Whatman no.1 filter paper and appropriately dilute with methanol. **Determination of total flavonoid contents** 1. Mix the plant extracts (0.5 mL) with 1.5 mL of methanol of 10% aluminum chlori mL of 1 M potassium ac and 2.8 mL of distilled w keep at room temperatu	**Solvent extraction using acetone** 1. Extract the *S. chinensis* fruit pulp (SCFP) by homogenizing in acetone by keeping fruit pulp to solvent ratio of 1:10. 2. Carry out the extraction on an orbital shaker for 24 h at room temperature. 3. Centrifuge at 10,000 rpm at 4 °C for 15 min. 4. Filter the homogenate through Whatman No. 1 filter paper and use. Determination of total flavonoid content hloride extracts, 1.5 .1 mL of 10% M potassium acetate and 2.8 mL of distilled	**Extraction and fractionation with butanol** 1. Collect fruits of *P. persica*. 2. Remove seeds from *P. persica* fruits (5 kg) and extract with 80% acetone in the ratio 1:10 w/v in the warring blender for 5 min and then homogenize by Polytron homogenizer for 3 min. 3. Process the left residue with the same procedure for three times. 4. Filter the combined extracts through Buchner funnel and evaporate under reduced pressure at 50 °C up to 90%. 5. Successively partition remaining liquid with hexane, ethyl acetate and n-butanol. 6. Pool the n-butanol extracts separately and evaporate to dryness under reduced
Concentration	4.75 ± 1.14 mg QE/g dry weight (Stem), 7.82 ± 0.67 mg QE/g dry weight (Leaf), 2.05 ± 0.49 mg QE/g dry weight (Fruit) View Less	0.20 ± 0.28 mg of rutin equivalents (RE) per gm fresh weight	28.26 ± 2.36 mg quercetin / g

分析手法やバリデーションの比較

バリデーション

PDF または Excel 形式でダウンロード可能

収録内容

2016 年 3 月現在

収録数	約 15 万件 （今後も収録拡大予定！）
収録期間	2000 年～
収録分野	医学分野，農学分野，化学分野を中心とし， その他周辺分野も収録
収録雑誌例	・Food Chemistry ・Journal of Chromatography A ・Journal of Chromatography B ・Journal of Agricultural and Food Chemistry ・Talanta ・Analytica Chimica Acta

検索例

【1. キーワード検索】
逆相クロマトグラフィによる錠剤中のラミブジンの分析方法

【2. カテゴリー検索】
天然物の単離方法

METHODSNOW™
A CAS SOLUTION

無料トライアル実施中！

契約については化学情報協会まで
お問合せください.

JAICI
化学情報協会

CAS
A DIVISION OF THE
AMERICAN CHEMICAL SOCIETY

Title of Method	Analysis of Efavirenz in Pharmaceutical tablets by Reversed-phase HPLC Link to Details	Analysis of Abacavir in Pharmaceutical tablets by Reversed-phase HPLC Link to Details
CAS Method Number	1-101-CAS-36158	1-101-CAS-24455
Method Category	Active Pharmaceutical Ingredient and Metabolite Analysis	Active Pharmaceutical Ingredient and Metabolite Analysis
Technique	Reversed-phase HPLC; Photodiodes	Liquid chromatographic UV detectors; Reversed-phase HPLC
Analyte	Azidothymidine; Efavirenz; Lamivudine	Abacavir; Lamivudine
Matrix	Pharmaceutical tablets	Pharmaceutical tablets
Other Materials	Reverse phase C18G, 250 × 4.6 mm; 5 µ column; Nylon membrane filter, 0.45 µm	Methanol; C_{18} column (250 mm × 4.6 mm i.d., 5 µm particle); Whatman filter paper no: 41
Equipment Used	HPLC system, e2695 Alliance, Waters PDA Detector, 2998 Electronic analytical weighing balance (0.1mg sensitivity), AY 220, Shimadzu Digital pH meter, DELUX 101 Sonicator, Sonica, model 2200 MH	HPLC system, Shimadzu UV-visible detector, SPD 20A, Shimadzu
Conditions	**Chromatographic Conditions** mobile phase: acetonitrile and potassium dihydrogen orthophosphate buffer in ratio of 30:70, v/v, flow rate: 1.0 ml/min **Instrument Conditions** detection: 275 nm	**Chromatographic Conditions** Mobile phase- MeoH : phosphate buffer (74.3 : 25.7% v/v) (pH 6.85, buffer strength 0.05 M); flow rate- 1.2 ml/min; injection volume- 20 µl **Instrument Conditions** Wavelength- 260 nm
Source	**RP-HPLC method development and validation for the simultaneous quantitative estimation of Efavirenz, Lamivudine and Zidovudine in tablets** Rajkumar, B.; Bhavya, T.; Kulsum, S.; Ashok Kumar, A. International Journal of Pharmacy and Pharmaceutical Sciences (2014), 2(6), 87-92, 6 pp. CODEN:IJPPKB ISSN:09751491 International Journal of Pharmacy and Pharmaceutical Sciences Document Sources Objective: To develop a new, simple, accurate, precise, linear and rapid Reverse Phase High Performance Liquid Chromatog. (RP-HPLC) method for the simultaneous quant. estimation of Efavirenz, Lamivudine and Zidovudine in tablets as per ICH guidelines. Methods: The optimized method uses a reverse phase C18 column, Enable C18G (250 × 4.6 mm;5µ), a mobile phase consisting of acetonitrile:0.02M potassium dihydrogen orthophosphate buffer adjusted to pH 3.2 in the proportion of 30:70 volume/volume, flow rate of 1.0 mL/min and a detection wavelength of 275nm using a UV detector. Results: The developed method resulted in Efavirenz eluting at 2.01 min, Lamivudine at 2.90 min and Zidovudine at 7.52 min. The linearity of the method was over the range of 75-450 µg/mL for Efavirenz, 18.75-112.5 µg/mL for Lamivudine and 37.5-225 µg/mL for Zidovudine. The method precision was exemplified by relative standard deviations of 0.15% for Efavirenz, 0.24% for Lamivudine and 0.37% for Zidovudine. Percentage Mean recoveries obtained during accuracy were in the range of 98-102. The limit of detection (LOD) was obtained as 20ng/mL for Efavirenz, 1ng/mL for Lamivudine and 2ng/mL for Zidovudine. The limit of quantitation (LOQ) was obtained as 50 ng/mL for Efavirenz, 2.5ng/mL for Lamivudine and 5ng/mL for Zidovudine. Conclusion: A new, simple, accurate, precise, linear and rapid RP-HPLC method was developed and validated for the simultaneous estimation of Efavirenz, Lamivudine and Zidovudine mg in tablets as per ICH guidelines. Hence the method can be used for the routine anal. in various pharmaceutical industries.	**Simultaneous optimization of the resolution and analysis time in RPHPLC Method for Abacavir and Lamivudine using Derringer's desirability function** Sudha, T.; Shanmugasundram, P. International Journal of PharmTech Research (2014), 3(6), 1040-1048 CODEN:IJPRIF ISSN:09744304 Sphinx Knowledge House Document Sources A High performance liquid chromatog. method has been developed and optimized for antiretroviral drugs (Abacavir and Lamivudine). Multiple response simultaneous optimization using the Derringer's desirability function was employed for the development of RP-HPLC. The possibilities of the simultaneous drug anal. allow a decrease of time during the assay and save reagents and solvents. The ranges of independent variables used for the optimization were MeOH (65-75%volume/volume), pH (6.0 - 7.0), flow rate (0.8 -1.2 mL/min). The influence of these variables on the output responses such as capacity factors of the first peak (k1), resolutions (Rs1,2) and retention time (tR2)were evaluated. The exptl. responses were fitted into a second order poly nominal and the three responses were simultaneously optimized to predict the optimum conditions for the effective separation of the studied components. Optimum conditions chosen for assay were MeoH: phosphate buffer (74.3:25.7%volume/volume) (pH 6.85, buffer strength 0.05M) and flow rate of 1.2 mL/min.The eluate was monitored using an UV detector set at 260 nm. Total chromatog. anal. time was approx. 5.0 min. The optimized assay condition was validated as per International Conference on Harmonization guidelines to confirm specificity, linearity, accuracy, limit of detection, limit of quantification and precision.

Preparation	**Buffer preparation** 1. Prepare the buffer solution by weighing 2.736 g of potassium dihydrogen orthophosphate (KH_2PO_4) and transfer to 1000 ml of HPLC grade water to get 20 mM buffer strength. 2. Adjust to pH 3.2 using 30% v/v ortho phosphoric acid. 3. Filter the buffer through 0.45 µm nylon membrane filter. **Mobile phase preparation** 1. Prepare the mobile phase by mixing acetonitrile and buffer in the ratio of 30:70, v/v. 2. Sonicate for 10 minutes for the removal of air bubbles. **Sample Preparation** 1. Prepare sample solution by dissolving tablet powder into diluents (mobile phase). 2. Weigh ten tablets separately and determine their average weights, and take in a 100 ml volumetric flask, dissolve in diluents and dilute up to 100 mL using mobile phase. 3. Sonicate for about 10 minutes then filter through 0.45 µ membrane filter to get standard stock sample solution. 4. Pipette out 5 mL of the above stock solution out and dilute to 100 ml to get a concentration, considered as working sample solution, 100% target concentration. **Standards Preparation** 1. Take 600 mg of Efavirenz, 150 mg of Lamivudine and 300 mg of Zidovudine in 100 ml volumetric flask containing 50 ml of diluent./li> 2. Sonicate and dilute using the mobile phase (consider this as standard stock solution). 3. Pipette out 5 ml of the above stock solution and dilute up to 100 ml to get a concentration, considered as working standard solution, 100% target concentration.	**Standards Preparation** 1. Weigh accurately 25 mg of Abacavir and 25 mg of Lamivudine and transfer into a 25 ml volumetric flask and dissolve with methanol, then dilute with the same (1 mg/ml). 2. Transfer 2.5 ml of the solution into 50 ml standard flask and dilute with mobile phase (50 µg/ml).
Method	**HPLC** 1. Perform the analysis using a Waters e2695Alliance HPLC system connected with PDA Detector 2998 and Empower2 Software. 2. Inject a volume of 20 µL sample solution. 3. Carry out the separation using a reverse phase C18 column, Enable C18G (250× 4.6 mm; 5 µ). 4. Use the mobile phase consisting of a mixture of acetonitrile and potassium dihydrogen orthophosphate buffer (20 mM, pH adjust to 3.2 using ortho phosphoric acid) in ratio of 30:70, v/v. 5. Set the mobile phase at a flow rate of 1.0 ml/min. 6. Set the detection wavelength at 275 nm. 7. Acquire the drug analysis data and process using Empower2 software running under Windows XP.	**Method or Procedure** 1. Weigh accurately twenty tablets, determine the average mass per tablet and finely powder. 2. Weigh accurately the powder equivalent to 25 mg of each and add a minimum quantity of methanol to dissolve the substance. 3. Dilute up to 25 ml with methanol (1000 µg/ml) in a volumetric flask. 4. Sonicate the solutions for 10 min, and filter through Whatman filter paper no: 41. 5. Separate out the insoluble excipients. 6. Collect the filtrate after rejecting the first portion of the filtrate. 7. Dilute 2.5 ml of the clear solution up to 50 ml with mobile phase to obtain 50 µg/ml. 8. Further dilute 3 ml to 10 ml with mobile phase to obtain 6 µg/ml. **Method or Procedure** 1. Inject 20 µl of sample onto Gemini C_{18} column (250 mm × 4.6 mm i.d., 5 µm particle). 2. Use MeoH : phosphate buffer (74.3 : 25.7% v/v) (pH 6.85, buffer strength 0.05 M) as a mobile phase. 3. Maintain the flow rate at 1.2 ml/min. 4. Set the UV-visible detector at 260 nm. 5. Analyze the data using chromatographic software Autochro 3000.
Linearity Range	75-450 µg/ml, Efavirenz; 18.75-112.5 µg/ml, Lamivudine; 37.5-225 µg/ml, Zidovudine	2 - 12 µg/ml, Lamivudine; 2 - 12 µg/ml, Abacavir
Limit of Detection	20 ng/ml, Efavirenz; 1 ng/ml, Lamivudine; 2 ng/ml, Zidovudine	0.0589 µg/ml, Lamivudine; 0.0012 µg/ml, Abacavir
Limit of Quantitation	50 ng/ml, Efavirenz; 2.5 ng/ml, Lamivudine; 5 ng/ml, Zidovudine	0.0204 µg/ml, Lamivudine; 0.0115 µg/ml, Abacavir
Accuracy	101.6, 99.4, 100.33% Recovery in 150, 300, 450 µg/ml added sample respectively, Efavirenz; 101.6, 99.2, 98.75% Recovery in 37.5, 75, 112.5 µg/ml added sample respectively, Lamivudine; 100.5, 100.93, 100.66% Recovery in 75, 150, 225 µg/ml added sample respectively, Zidovudine	99.13%, Lamivudine; 100.41%, Abacavir
Precision	0.49% RSD (Inter-day), Efavirenz; 0.50% RSD (Inter-day), Lamivudine; 0.57% RSD (Inter-day), Zidovudine	0.57% RSD, Lamivudine; 1.05% RSD, Abacavir
Retention Time	2.02 min, Efavirenz; 2.90 min, Lamivudine; 7.52 min, Zidovudine	3.74 min, Lamivudine; 3.5 min, Abacavir