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A Review on a Some Analytical Methods for Determination of Salicylic Acid

Amina M. Abass[1*], Jamal M. Rzaij[2], Huda ghalib Salman[1], Wisam Kadhum H.Al-Hashemi[1]

[1]Department of Chemistry, College of Science, Al-Nahrain University, Al-Jaderia, Baghdad-Iraq
[2]Department of Physics, College of Science, University of Anbar, Ramadi, Iraq

***Corresponding Author:** Amina M. Abass, Department of Chemistry, College of Science, Al-Nahrain University, Al-Jaderia, Baghdad-Iraq. Email: aminamohsen75@gmail.com*

ABSTRACT

Salicylic acid is being used as a keratolytic agent for treatment of corns or verrucae; it's being used in many ointments and medicines due to its ability to remove the stratum corneum. Since such products containing salicylic acid are easily obtainable, salicylic acid poisoning (salicylism) cases are relatively many especially for infants not older than 5 years. Its poisoning symptoms were reported to be headache, vertigo, tinnitus, bradyacusia, amblyopia and sweating. There are many analytical techniques that have been reported for simultaneous estimation of salicylic acid and its derivative pharmaceutical dosage form such as : Aspirin, acetylsalicylic acid, salicylamide, sodium salicylate, p-amino salicylic acid and methyl salicylate. Some of those techniques are UV Spectrophotometry, high-performance liquid chromatography (HPLC),liquid chromatography - mass spectrometry (LC-MS), gas chromatography (GC), and ultra-performance liquid chromatography UPLC), In addition of various analytical methods are available for the quantification of pharmaceutical forms, ion-Selective Electrodes one of the most important chemical technique which used for determination of salicylic acid in pure and pharmaceutical formations.

***Keywords:** ISEs, Salicylic acid, Review, Analytical Methods. Spectrophotometry, HPLC*

INTRODUCTION

Salicylic acid is chemically 2-hydroxy benzoic acid that has antiseptic, antifungal and keratolytic properties. It is used to treat warts, psoriasis, corns and other skin conditions. It works by softening and loosening dry, scaly, or thickened skin so that it falls off or can be removed easily [1,2].

Salicylic acid is white or almost white, crystalline powder or white or colorless, acicular crystals, slightly soluble in water, freely soluble in ethanol, sparingly soluble in methylene chloride, Its molecular formula is $C_7H_8O_3$ and molecular weight is138.1 gm $mole^{-1}$. Its melting point is 158-161^{0}C., chemical structure shown in Figure 1[3].

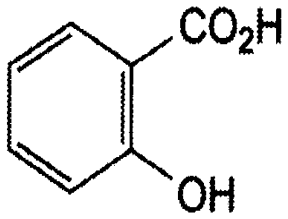

Figure1. Chemical structure of Salicylic acid

In this literature review, various analytical methods are demonstrated for salicylic acid, some of these techniques are reported and it is given as Table1, Table 2 and Table 3

Table1. *Spectro photometric methods for determination salicylic acid*

Name of Method	Results	Ref. No
UV-Spectral absorption by change in PH	**Sample** :Aspirin in blood λ_{max}**used**=300, 319 nm **Range of acetylsalicylic acid**=9.3-10.2 mg/µ.,with **SD**= ±0.32 **Range of salicylic acid**=22.2-23.7 mg/µ.,with **SD**= ±0.77	**4**
UV-Visible Spectrophotometric	**Sample :**salicylic acid λ_{max} **used**=300 and 250 nm. **Concentration Range**=5-50µg/ml **%Drug content in marketed formulation**=103.85	**5**
PH-Selective Visible Spectrophotometric	**Sample** :salicylic acidas a chromogenic agent λ_{max} **used**=520 , 460 nm.	**6**

	PH=2.26(±0.02),**PH**=6.1(±0.02) **Concentration Range**=3.75-37.5µg/ml and 2.0-24.0 µg/ml. **R**=0.999,**R**=0.9988 **Apparent molar absorptivity** =1.7×10^3, 3.01×10^3 L mol^{-1} cm^{-1}	
Kinetic Spectrophotometric	**Sample** :acetylsalicylic acid λ_{max} **used**=410 nm. **Concentration Range**=0.72-9.00 µg/ml **Detection Limit**=0.35 µg/ml	**7**
Spectrophotometric by Solvent Extraction	**Sample** :salicylic acid λ_{max} **used**=516 nm **Concentration Range**= 8×10^{-6}- 4×10^{-5}M **PH**=5.5-9.	**8**
Absorptiometric in Aqueous Ethanolic Solution	**Sample :** acetylsalicylic acid λ_{max} **used**=276,300 nm. **Molar absorptivity** =1.1×10^3, 3.83×10^3 L mol^{-1} cm^{-1}	**9**
Spectrophotometry	**Sample :** salicylic acid λ_{max} **used**=303 nm. **Solvent:** ethanol **%Recovery** =99.5-101.3	**10**
Spectrophotometric Analysis	**Sample :** salicylic acid **Solvent:** ethanol, chloroform, benzene, etc. **Mean±S.D**=100.99±0.9 **Coefficient of Variation**=0.989 **Std. error** =0.577	**11**
Spectro fluometry	**Sample:** salicylamide, acetylsalicylic acid, and salicylic acid **Concentration Range:** 10^{-7}M	**12**
First Order Spectroscopy	**Sample :**Aspirin λ_{max} **used**=234.15-238.88 nm **Solvent :**Methanol **Amplitudes=** 232.98nm **Concentration Range**:2-10 µg mL^{-1} **%Assay commercial formulation**=98.74-101.24	**13**
Spectrophotometric	**Sample :**Sodium salicylate λ_{max} **used**= 452 nm **Concentration Range**= 2-30µg.ml^{-1} **Molar absorptivity** =0.0188 µg/cm^2,8.5013×10^3 L mol^{-1} cm^{-1}	**14**
UV – Visible Spectrophotometer	**Sample :**Aspirin λ_{max} **used**= 220 nm **Solvent**=ethanol **%Recovery**=97.70,98.73,96.85,99.75,98.22	**15**
Second Derivative Ultraviolet Spectrometry	**Sample :**Aspirin λ_{max} **used**= 292 nm **Solvent**=chloroacetic acid-ethanol **Concentration Range**=1.00-10.02µg.ml^{-1} **R**= 0.9999 **%RSD**= 1.2	**16**
Spectrophotometry	**Sample**: p-Amino salicylic Acid λ_{max} **used**= 460-555nm **Buffer solutions**=3M (HCl-KCl), 5M (HCl-KCl) **Concentration Range**=0.4-2.0 µg.ml^{-1} **Molar absorptivity**=2.4×10^4 and 3.8×10^4 L mol^{-1} cm^{-1} λ_{max} **used**= 264 nm **Molar absorptivity**=7.65×10^3 L mol^{-1} cm^{-1} **Concentration Range**=2-10 µg.ml^{-1} **%Recovery**=97.6±1.71,98.4±1.45	**17**
Spectrophotometry	**Sample:** salicylic acid λ_{max} **used**= 540 nm **Solvent** :water ,chloroform ,amyl alcohol ,ether ,ethanol **P-value**=0.993 , 0.000	**18**

Table2. *High-performance liquid chromatography For Determination of salicylic acid*

Name of Method	Results	Ref.No

LC-MS/MS	**Sample:** salicylic acid **PH**=4.2 **Concentration Range**=1-1000 ngL^{-1} **%Re**=95-100 $\mathbf{R_{(n=6)}}$=0.99	**19**
Liquid Chromatography	**Sample:** salicylic acid **PH**=3.0 **Concentration Range**=0.1-50$\mu g.ml^{-1}$ **R**=0.999 **%Re**=95.6-103.9 **%RSD**=3.8	**20**
High-performance liquid chromatography with Gas chromatography	**Sample**=methyl salicylate and salicylic acid **Concentration Range**=0.07-0.89 $\mu g.ml^{-1}$ **Concentration Range**=1.32-6.54 $\mu g.ml^{-1}$	**21**
High-Performance Liquid Chromatographic	**Sample :**Aspirin, salicylic acid **Concentration Range**=0.5-1.25 $\mu g.ml^{-1}$, 0.4-6.0% $\mu g.ml^{-1}$ **%Re=100.2,99.2** **Coefficient of variation** $_{(n=6)}$ =0.3,2.6. **Relative retention time**=3.6,6.4 sec.	**22**
Reversed phase liquid Chromatography	**Sample:** acetylsalicylic, salicylic acid **Concentration Range**=0.21-0.39 $\mu g.ml^{-1}$,6.3-11.7 $\mu g.ml^{-1}$ **R**=0.9995,0.9988 **Detection Limit**=0.23 $\mu g.ml^{-1}$,0.69 $\mu g.ml^{-1}$ **Sensitivity**=1.88mAbs($\mu g.ml^{-1}$),1.84mAbs($\mu g.ml^{-1}$)	**23**
GC-MS	**Sample:** salicylic acid, methyl salicylate **%RSD**=1.22,1.67 **R=0**.9976 to 0.9997	**24**
UPLC-MS/MS-API-5500	**Sample**: Aspirin, salicylic acid **Concentration Range**=0.1-24 ng/ml,0.1-8 $\mu g.ml^{-1}$ **%Mean Recovery** = > 85%	**25**
Novel HPLC	**Sample:** salicylic acid **Detection Limit**=0.125,0.250 $\mu g.ml^{-1}$ **%Re**=98.99±0.66 , 97.50±1.33	**26**
RP- HPLC	**Sample:** salicylic acid λ_{max} **used**= 263 nm **Buffer**=(55:45) **Concentration Range**=20-80 ppm **Flow Rate**=1ml/min **%Re**=99.46	**27**
HPLC Profiles	**Sample:** salicylic acid**,** acetylsalicylic **λUV-Detection** = 280-360 nm **Flow Rate**=1.0 ml/min **Runtime**=22 and 25 min at solvent methanol and acetic acid**.** **Runtime**=30 and 45 min at solvent acetonitrile and phosphoric acid	**28**
LC-MS/MS	**Sample:** salicylic acid **Concentration Range**=5.30-12.8 mg/kg and 0.13-1.01 mg/kg **R**= 0.9911 to 0.9936 **%Re**=98.3 to 101	**29**
Liquid-Liquid Extraction and High-Perphormance Liquid Chromatography	**Sample:** salicylic acid **Concentration Range**=5-10 $\mu g.ml^{-1}$ **Detection Limit**=0.0048ng/ml **%Re**=88.0-95.0 **%RSD**=3.8 to 6.8	**30**
UV-HPLC	**Sample:** Aspirin, salicylic acid λ_{max} **used** = 254 nm for HPLC,226,296 nm for UV. **Solvent**=acetonitrile and ratio of water for UV and used as a mobile phase in HPLC.	**31**

	R^2=0.9996, 0.9992 in UV, 0.006,0.004 µg.ml^{-1} for HPLC. **%Re**=98.80 to 101.26 and 98.67 to 103.33	
HPLC	**Sample:** Aspirin **%Re**=91.0-97.8 ,94.3-102.7 ,92.3-107.6 ,102.5-103.6 **%RSD**=0.4 ,0.2 ,0.5 ,0.6 ,3.4 R^2=0.997 ,0.999 ,0.999 ,0.995 ,0.998	**32**
HPLC Analysis	**Sample:** salicylic acid λ_{max} **used** =334 nm **Concentration Range=** 0.038–0.56 mg/mL **Solvent:** acetonitrile-acetic acid **Run**= 1 mL/min	**33**
Reverse phase high performance liquid chromatographic	**Sample:** salicylamide, salicylic acid λ_{max} **was used** =245 nm **Concentration Range=**1-50 µg.ml^{-1} R^2=≥ 0.99 **%RSD=** less than 1.0 **%Re**=99.74, 99.52 ,99.40	**34**
HPLC and GC/MS	**Sample:** salicylic acid , acetylsalicylic acid **PH**=9.0 **Net mobility**=6.3×10^{-4} cm^{2}V^{-1} S^{-1} ,5.6×10^{-4} cm^{2}V^{-1} S^{-1} **Electro –osmotic mobility**=9.3×10^{-4} cm^{2}V^{-1} S^{-1}	**35**
Liquid chromotography	**Sample:** Salicylic Acid λ_{max} **was used**= 245 nm **Concentration Range**=256-384 µg/ml **%Re**=100.56, 100.63 , 100. **Flow rate**=1.5 ml/min	**36**

Table3. *Ion –Selective Electrodes for Determination salicylic acid*

Type of Ion –pair for Electrodes	Slope mV/decade	Conc. Range	Detection Limit	R	Response Time	PH	Life Time	Ref.
[bis(2-hydroxyl imino)1-phenyl,2-(2-qunolile) 1-ethanona] Aluminium(III)	–59.1 ± 0.4	1.0 × 10^{-6}-1.0 × 10^{-1} (mole/L)	5.0 × 10^{-7} (mole/L)	0.998	15 sec	2.0–7.8	-	**37**
Zinc aluminium layered double hydroxides-4(2,4-dichlorophenoxy) butyrate (Zn/Al-DPBA) nanocomposite	58.8 ± 1.0	1.0 x 10^{-5}-1.0 x 10-1 (mole/L)	3.9 x 10^{-6} (mole/L)	-	11-35 sec	4.0-12.0	4 months	**38**
Trioctylmethyl ammonium chloride	56±1.0, 57.7±1.0	3×10^{-6}–1 1×10^{-6}–1 (mole/L)	8×10-^{7} (mole/L)	-	~ 5 sec	5.0-12.0	-	**39**
Second kind Pt|Hg|Hg$_2$(salic)$_2$ Graphite	58.66	6.0×10^{-4}-1.0×10^{-1} (mole/L)	-	-		6.0	18 months	**40**
ZnO/Al2O3 nanocomposite	-	0.5-80.0 µM.	0.25 µM	-	-	2.0-9.0	-	**41**
Pencil graphite electrode	29.60 ± 0.30	1.0 × 10^{-5} - 1.0 × 10^{-1} (mole/L)	5.42 µM	-	<20 sec	4.0-6.0	-	**42**
Ferric(III) nitrate and copper(II) acetate on Dowex-50x8	-	0.02–0.50 g/L 0.40–1.40 g/L	-	-	10,8 min	2.1 ,6.5	-	**43**
Acetylsalicylic Acid in Tablets with Salicylate Ion Selective Electrode in a Batch Injection Analysis System	43±4 and 45±2.5	7.5 x 10^{-3}–7.5 x 10^{-2} (mole/L)	-	-	< 2 sec	-	-	**44**
Calix[4]arene	58.8±0.5	1.0×10^{-5}–1.0×10^{-1} (mole/L)	4.3×10^{-6} (mole/L)	-	5–10 sec	4.0	-	**45**

Tubular salicylate	56.0	0.05--10 mM	0.05 mM	-	-	-	-	**46**
Glassy carbon electrode (GCE) modified with a composite film of poly (4–vinylpyridine) (P4VP) and multiwall carbon nanotubes (MWCNT)	-	0.04–350 µM	4.42 nM	-	-	7.4	-	**47**

CONCLUSION

It becomes necessary to analysis of drugs and selections of solvents that are greater challenges for the analysis. From this review of literature, It's found that commonly used diluents are ethanol, methanol, acetonitrile, phosphate buffer or distilled water in HPLC methods, which extended the run time with greater tailing factor. While, for Spectrophotometry estimation, presence of excipients that have multi–component dosage forms, produce significant challenge to the analyst during the development of assay. Estimation of individual drugs in multi component dosage forms becomes difficult. For multi component dosage forms, chemo-metric methods can be preferred to routine spectrometric methods. A very simple and easy method to determine Salicylic acid depends on Ion-Selective Electrodes. A systematic review of various analytical methods for determination of Salicylic acid or its derivatives for pharmaceutical dosage forms. Wide ranges of instrumental methods for quantitative estimation of Salicylic acid have developed successfully. However, the methods are time consuming and complex. A vast number of HPLC methods have been developed for analysis of Salicylic acid and its derivatives. For analysis of Salicylic acid in pharmaceuticals, HPLC with UV, ISEs detection are applicable because these methods provide simple, precise, rapid, accurate, and economical analytical methods for estimation of this drug.

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