JETIR.ORG ISSN: 2349-5162 | ESTD Year : 2014 | Monthly Issue
JOURNAL OF EMERGING TECHNOLOGIES AND INNOVATIVE RESEARCH (JETIR)
An International Scholarly Open Access, Peer-reviewed, Refereed Journal

Quantitative Analysis of Pharmaceutical Using FTIR Spectroscopy

Prasannjeet S. Bhopale [1]* Priyanka J. Anbhule[2], Aditi S. Awati[2], Sagar L. Chavan[2], Rutuja J. Patil[2]
Department of Pharmaceutical Chemistry, Bharati Vidyapeeth College of Pharmacy, Kolhapur 416013, Maharashtra, India

ABSTRACT:

Acetylsalicylic acid (ASA) is widely used globally to treat pain, rheumatic fever, and inflammation for over a century. It is also a prototypical molecule classified as a platelet aggregation inhibitor that could be widely used to minimize the risk of arterial and venous thrombosis in long-term therapy. There are numerous ASA formulations on the market and estimation of their quantity and efficacy is of utmost importance since it is largely being produced by many pharmaceutical companies all over the world. Literature is supported by many analytical methods using UV-visible spectrophotometer, liquid chromatography, liquid chromatography integrated with mass spectrometer (Lc-MS), UHPLc-MS/MS, Gas chromatography, electrochemical and titrimetric methods. In this study, an attenuated total Reflectance Fourier transform Infrared Spectroscopy (ATR-FTIR) method was developed for the estimation of ASA. The calibration curve was constructed on peak height location in the range of 2200-2500 cm^{-1} in the concentration range from 1-10µg/Ml with a correlation coefficient of 0.965. This new method was used for the quantification of ASA in tablets. During the spectral examination, excipients in the tablet formulation did not significantly interfere with the results.

Keywords: Aspirin, quantification, FTIR spectroscopy, assay

Introduction:

Infrared spectrometry (IR) provides a useful way for the identification of drugs. However, traditional techniques for obtaining IR spectra, such as alkali halide disks, mulls, and thin films, are not always adequate for quantitative analysis. FT-IR allows for the simultaneous analysis of several sample components and the ongoing observation of the spectral baseline. [1]. Acetylsalicylic acid (ASA, o-acetoxy benzoic acid, aspirin) (see Figure 1) is one of the oldest analgesic substances. Already Hippocrates (460-370 B.C.) used an extract of bark of willow trees containing a high quantity of salicylic acid to relieve pain. Aspirin is employed for alleviating mild to moderate pain, lowering fever, reducing redness and swelling, and aiding in the prevention of blood clot formation. It is used to relieve discomfort caused by numerous medical problems, including

headaches, infections, and arthritis [2]. It is also employed to minimize the likelihood of experiencing another heart attack or stroke. Larger doses of aspirin are used to treat gout. Acetylsalicylic acid inhibits the body's production of prostaglandins. Prostaglandins are hormone-like substances that are involved in the regulation of varied processes such as pain, fever, inflammations, and thrombosis. Prostaglandins elicit signals of pain and so by obstructing their synthesis, no pain can be felt. The techniques described for the assay of ASA are high-performance liquid chromatography, and other techniques relying on spectrophotometry or capillary electrophoresis techniques [3]. Analyzing drugs using FT-IR spectrometry yields a wealth of spectroscopic data on the major constituents present in the sample. [4].

2.1 Drug solubility problem & its effect on biological activity.

- It has a limited solubility in water, which amounts to 2–4mg/mL, and its solubility varies significantly with temperature [5-11].
- Aspirin is more soluble in ethanol, ethyl ether, chloroform, sodium hydroxide solution, and sodium carbonate solution than in water.
- Thesolubilityofaspirininthesesolventsisapproximately80,41, and 30 mg/ml, respectively.

a. According to pharmacological classification, aspirin belongs to the non-steroidal anti-inflammatory drugs (NSAIDs)and possesses a wide array of beneficial pharmacological activities which include antipyretic, analgesic, anti-inflammatory, and antiplatelet activities [12-15].

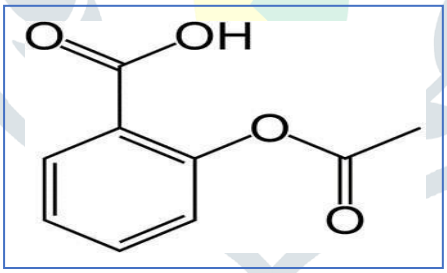


Figure1: Aspirin structure

Formula-$C_9H_8O_4$

Molecular wt.–Average:180.15

Melting point-135°C

MATERIALS AND METHODS

Material:

A. Chemicals and Pharmaceuticals: Aspirin was obtained as a gift sample from Medispray Laboratories, Satara. All the other chemicals were of analytical grade. Ecosprin tablet strips were purchased from a local chemist shop.

B. Infrared Spectroscopy: Bruker Alpha-II FTIR spectrophotometer was used for spectra measurement. Accessory: ATRecoZnSe#2014FEF82D, software: OPUX. The parameters were as follows:

Sr.No.	Parameter	Value
1.	Resolution	$4cm^{-1}$
2.	Sample scan frequency	24 scans
3.	Background scan frequency	24scans
4.	Spectrum range	$4000\text{-}600cm^{-1}$
5.	Interferogram size	15142points

Method:

A. Preparation of solution.

- **STEP1** • Preparation of stock solution of pure ASA and then serial dilution in the range1-10µg/mL
- **STEP2** • Preparation of stock solution of ASA tablet (Ecosprin)and then serial dilutionintherange1-10µg/mL
- **STEP3** • Measurement of FTIR spectra for each sample
- **STEP4** • Setting of new quant method in FTIR for plotting of calibration curve
- **STEP5** • Measuring the sample of ASA with unknown concentration using quantitative analysis
- **STEP6** • Reporting the result

B. Plotting of calibration curve

1. Thoroughly clean all the required glassware with acid and then wash two to three times with fresh distilled water.

2. Take 20 ml ethanol in a suitable beaker. Take 200 ml of fresh distilled water in a suitable beaker. Mix the component stock and prepare a 1:10 ratio of ethanol: water.

3. Weigh the 20 tablets of ecosprin of 75mg and calculate the average weight.

4. Triturate the aspirin tablets with the help of a mortar & pestle.

5. Prepare a stock solution of pure aspirin with a concentration of 100µg/mL using 1:10 ratio of ethanol: water. From this stock prepare serial dilutions in the range of 1-10 µg/ml

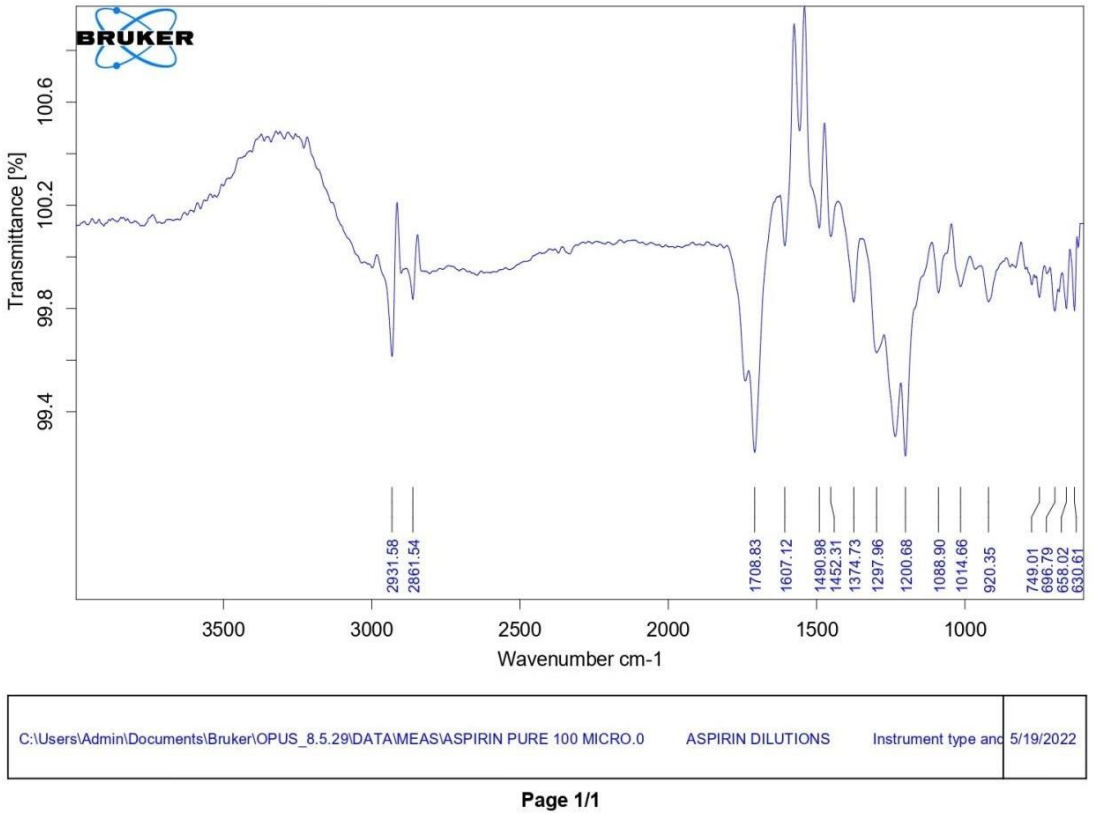
6. Similarly prepare stock solution of aspirin tablet with a concentration of 100µg/mLusing1:10 ratio of ethanol: water. From this stock prepare serial dilutions intherange1-10 µg/mL

7. Measure FTIR spectra for all the dilutions and save the files in the OPUX software. The integration method was set at a wavenumber of 1550-1750cm^{-1}.

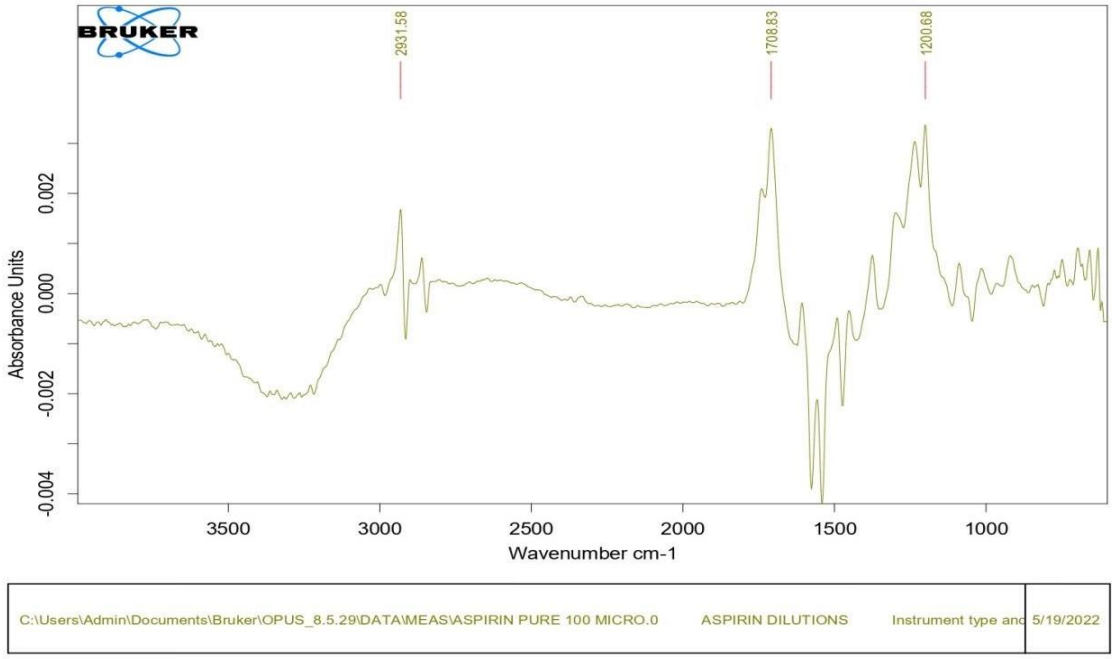
Due to variations in the relative peak strength of the absorption bands brought about by the internal reflection mechanism of ATR accessories, ATR spectra cannot be utilized using a quantitative approach designed using transmission spectra.

RESULT

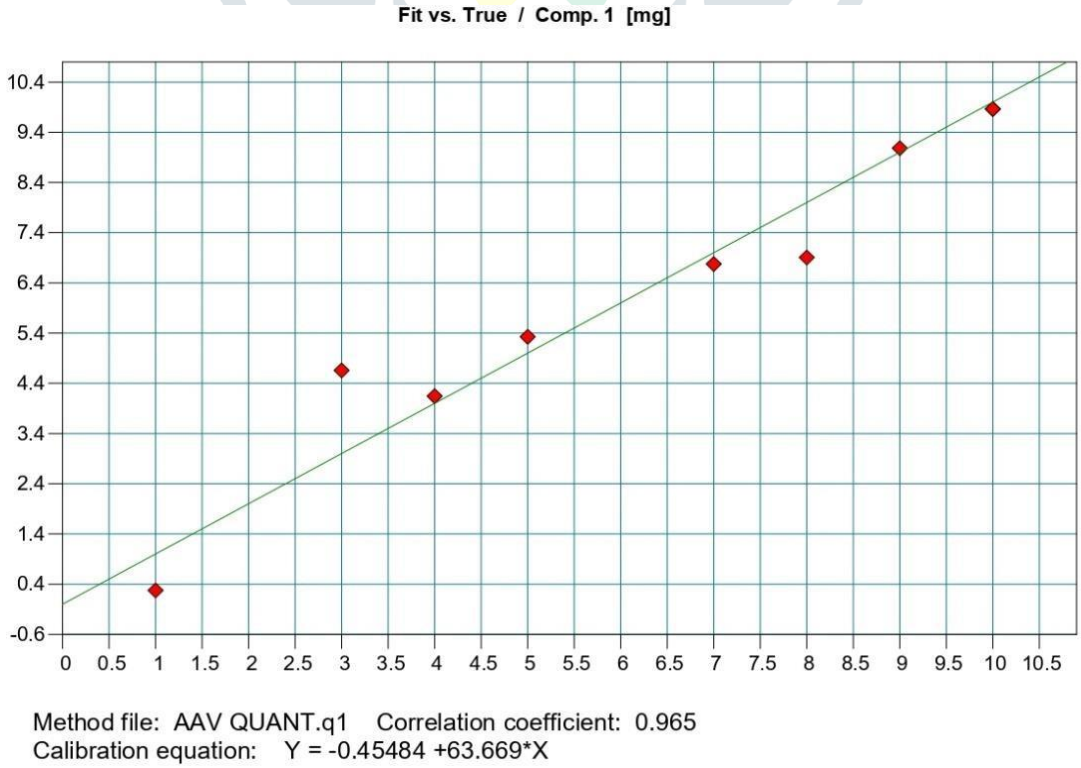
6.1 FTIR spectrum of pure aspirin powder (%transmittance)



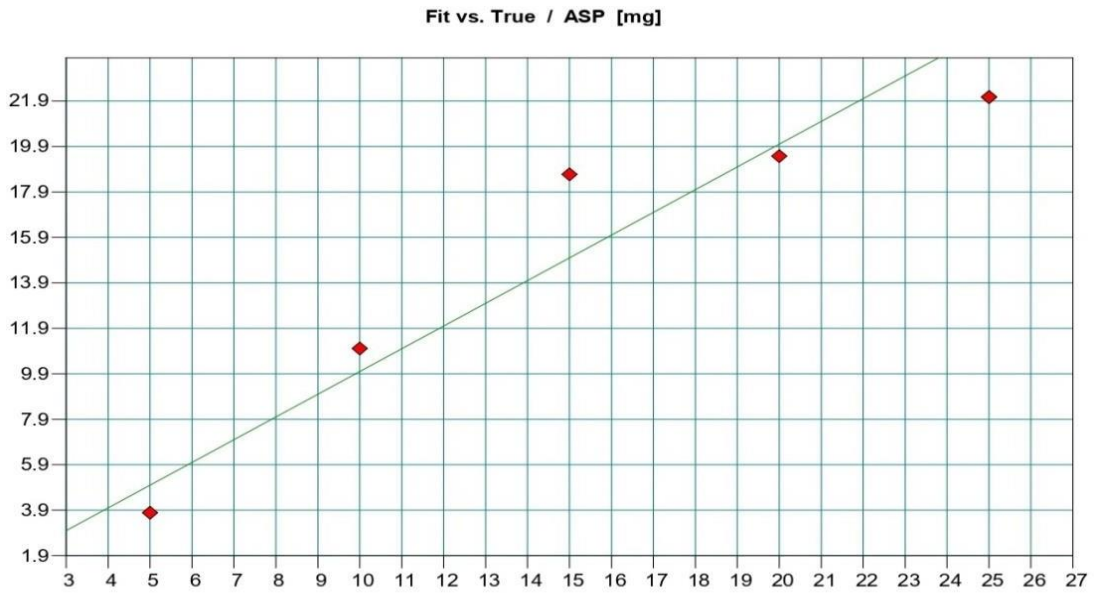
6.2 FTIR spectrum of pure aspirin powder (absorbance)



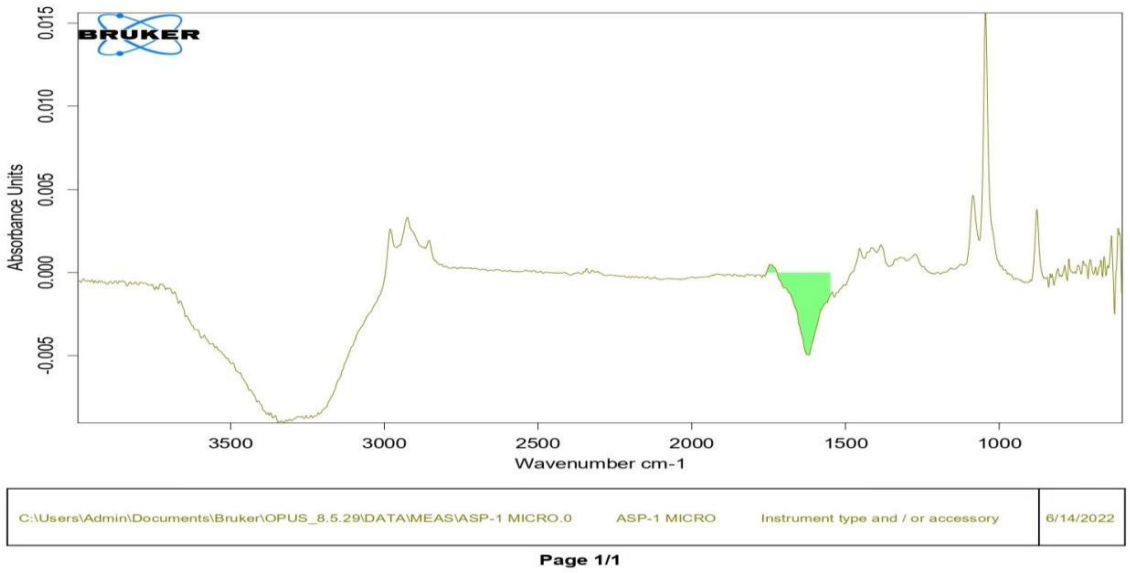
6.3 Calibration curve plot of pure aspirin in the range of 1-10µg/mL

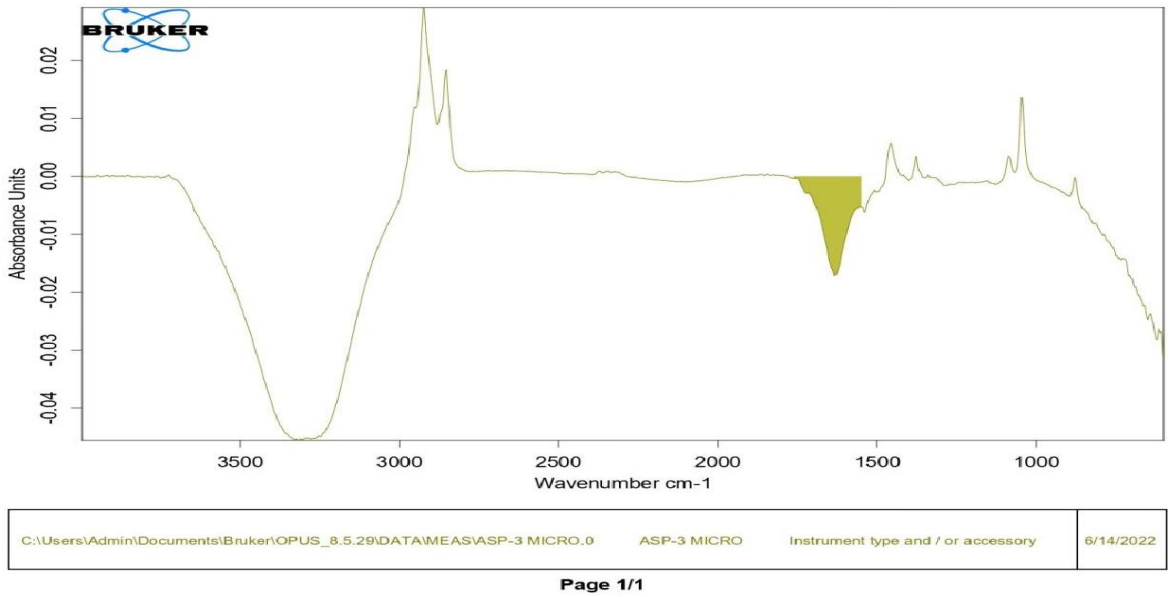


6.4 Calibration curve plot of tablet aspirin in the range of 1-10µg/mL

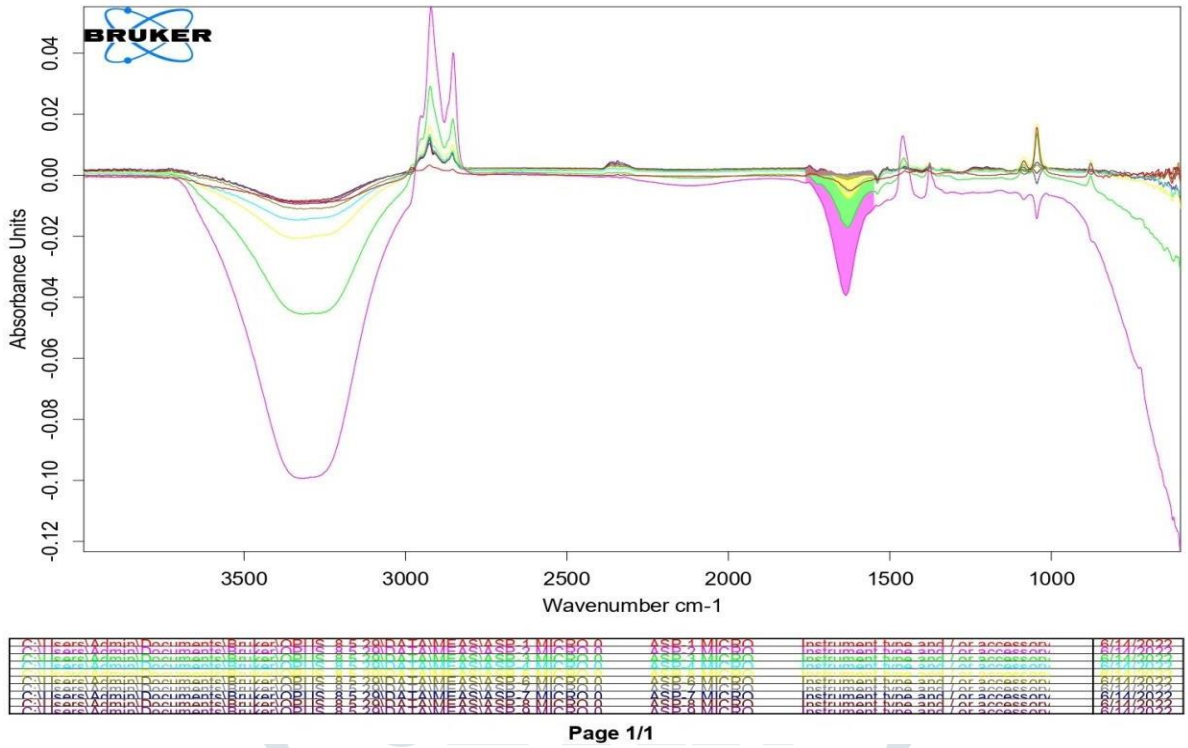


Method file: PRATIK.q1 Correlation coefficient: 0.949
Calibration equation: Y = +10.753 +13.146*X





6.5 Overlay of aspirin in the range of 1-10µg/mL



6.6 Estimation of the concentration of an unknown sample of aspirin

The concentration of unknown samples of aspirin from pure powder as well as the tablet was determined based on the above calibration curves and the concentrations of the samples are as follows:

6.6.1 Result for prediction of concentration of an unknown sample of pure aspirin:

Result of QUANT evaluation:

Spectrum file: C:\Users\Admin\Documents\Bruker\OPUS_8.5.29\DATA\MEAS\ASP-unknown MICRO.0Date and time (measurement): 14/06/2022 15:27:32.040 (GMT-7)
Method file: AAV QUANT.q1 - 2022/06/15 10:43:09 (GMT+5.5)

Component	Prediction	Sigma	Unit	Integr. Result
unkown	5.32	0.88	mg µg/mL	0.09

Signature (Operator) Signature (Release)

6.6.2 Result for prediction of concentration of an unknown sample of aspirin in tablet:

Result of QUANT evaluation:

Spectrum file: C:\Users\Admin\Documents\Bruker\OPUS_8.5.29\DATA\MEAS\ASP-unknown tablet.0Date and time (measurement): 14/06/2022 15:37:32.040 (GMT-7)
Method file: VAA QUANT.q1 - 2022/06/15 10:58:09 (GMT+5.5)

Component	Prediction	Sigma	Unit	Integr. Result
unkown	7.52	0.98	µg/mL	0.02

Signature (Operator) Signature (Release)

Sr.No	Unknown sample	Predicted value of concentration	Theoretical value of concentration	Standard deviation
1	Pure aspirin	5.32µg/mL	5µg/mL	0.2262
2	Tablet	7.52µg/mL	7µg/mL	0.3676

7. CONCLUSION

This proposed method expands the functionality of a conventional FTIR spectrophotometer, commonly utilized for identification purposes, to accurately quantify aspirin. Moreover, this method is less time-consuming, eco-friendly, and accurate. This method now enables the potential use of FTIR spectroscopy for quantifying active ingredients beyond aspirin.

8. REFERENCE:

1. Bunaciu AA, Aboul-Enein HY, Fleschin Ş. FT-IR Spectrophotometric analysis of acetylsalicylic acid and its pharmaceutical formulations. Canadian journal of Analytical sciences and spectroscopy. 2006 Jan 1;51(5):253-9.

2. Evans G, Packham MA, Nishizawa EE, Mustard JF, Murphy EA. The effect of acetylsalicylic acid on platelet function. The Journal of experimental medicine. 1968 Nov 1;128(5):877-94.

3. Bunaciu AA, Aboul-Enein HY, Fleschin S. Application of Fourier transform infrared spectrophotometry in pharmaceutical drugs analysis. Applied Spectroscopy Reviews. 2010 May17;45(3):206-19.

4. Karabacak M, Kose E, Kurt M. FT-Raman, FT-IR spectra and DFT calculations on monomeric and dimeric structures of 5-fluoro-and 5-chloro-salicylic acid. Journal of Raman Spectroscopy. 2010 Sep;41(9):1085-97.

5. Vane JR, Botting RM. The mechanism of action of aspirin. Thrombosis research. 2003 Jun15;110(5-6):255-8.

6. Elwood PC, Gallagher AM, Duthie GG, Mur LA, Morgan G. Aspirin, salicylates, and cancer. The Lancet. 2009 Apr 11;373(9671):1301-9.

7. Patrono C, García Rodríguez LA, Landolfi R, Baigent C. Low-dose aspirin for the prevention of atherothrombosis. New England Journal of Medicine. 2005 Dec 1;353(22):2373-83.

8. Jack DB. One hundred years of aspirin. The Lancet. 1997 Aug 9;350(9075):437-9.

9. Thun MJ, Namboodiri MM, Calle EE, Flanders WD, Heath Jr CW. Aspirin use and risk offatal cancer. Cancer research. 1993 Mar 15;53(6):1322-7.

10. Paikin JS, Eikelboom JW. Aspirin. Circulation. 2012 Mar 13;125(10):e439-42.

11. Fuster V, Sweeny JM. Aspirin: a historical and contemporary therapeutic overview. Circulation. 2011 Feb 22;123(7):768-78.

12. Gasparyan AY, Watson T, Lip GY. The role of aspirin in cardiovascular prevention: implications of aspirin resistance. Journal of the American College of Cardiology. 2008 May13;51(19):1829-43.

13. Langley RE, Burdett S, Tierney JF, Cafferty F, Parmar MK, Venning G. Aspirin and cancer: has aspirin been overlooked as an adjuvant therapy?. British journal of cancer. 2011 Oct;105(8):1107-13.

14. Bond AD, Boese R, Desiraju GR. On the polymorphism of aspirin. Angewandte Chemie International Edition. 2007 Jan 15;46(4):615-7.

15. Samter M, Beers Jr RF. Concerning the nature of intolerance to aspirin. Journal of Allergy. 1967 Nov 1;40(5):281-93.

16. Chandarana C, Kapupara P. Quantitative Simultaneous Estimation of Aspirin and Omeprazole by rp-Hplc Method Implementing Aqbd Approach in Pharmaceutical Dosage Form. Risk management. 2017;6:10.

17. Acharya S, Daniel A, Gyadangi B, Ramsamy S. Isolation, characterization of a potential degradation product of aspirin and an HPLC method for quantitative estimation of its impurities. Journal of chromatographic science. 2015 Oct 1;53(9):1491-7.

18. Patel D, Patel N, Vaishy R, Patel V, Solanki C, Patel M. Development and validation of RP- HPLC method for simultaneous estimation of aspirin and esomeprazole magnesium in tablet dosage form. Journal of Chemistry. 2013 Jan 1;2013.

19. Sharma M, Pathak M, Roy B, Jain L, Yadav N, Sarkar B, Kaur M, Sharma M. Quantitative estimation of aspirin in various drugs: UV-Vis absorption spectroscopy and colorimetric studies. DU J Undergraduate Res Innovation. 2010;4:157-62. Maheshwari RK, Saxena M, Gahlot M, Chaki R, Kinariwala M, Jagwani Y. Novel applicationof hydrotropic solubilizing additives in the estimation of aspirin in tablets. Indian journal of pharmaceuticalsciences.2010Sep;72(5)