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Isolation, Identification and Characterization of Afatinib Novel Degradation Products by NMR and HRMS:RP-UPLC Method Development and Validation

Ramulu Yanaka[1, 2,*], Hima Bindu Gandham[2], Chidananda Swamy Rumalla[1], Muralidharan Kaliyaperumal[1] Shaik john Saida[1], Rudrakshula J D Prasad[1], and Raghu nandan Renukunta[1]

1. Department of Medicinal Chemistry, GVK Biosciences Pvt. Ltd, IDA Mallapur, Hyderabad, Telangana-500076, **INDIA**
2. Department of Engineering Chemistry, Andhra University, Visakhapatnam-530003, A.P., **INDIA**

Email: ramu.yanaka@gmail.com, chidanand_swamy@yahoo.co.in

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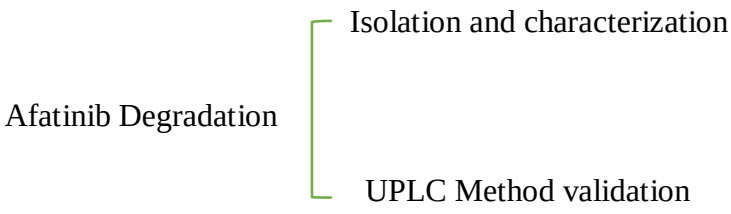
ABSTRACT

Afatinib (AFA) is an anilino quinazoline derivative and it was subject to stress degradation under acidic, basic, peroxide mediated oxidation, photolytic and thermal degradation. The stress degradation was performed according to ICH guidelines Q1A(R2) and the drug was inert under thermal and photolytic conditions. Two degradants were identified in acid hydrolysis referred as AFA-DP1, AFA-DP2 , one degradant was formed in base hydrolysis referred as AFA-DP3 and one degradant was formed in peroxide mediated hydrolysis referred as AFA-DP4. Out of four degradants two are novel and two are already published, here also one degradant structure was conformed by mass and another one by 1H and 13C NMR. In our study all the four degradation product structures were confirmed by HRMS and 1D (1H, 13C) and 2D (COSY, HSQC and HMBC) based on 1D and 2D NMR data proton and carbon chemical shift values assigned exactly for all DPs. A stability indicating RP-UPLC method was developed and validated with shorter run time and method was validated in terms of linearity, specificity, accuracy, LOD and LOQ.

Graphical Abstract



Keywords: Afatinib, Method validation**,** Degradation products, HRMS, NMR, ^{15}N HSQC, ^{15}N HMBC.