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Impurity Profile and Validation of Benzbromarone and Diclofenac Combined Drug by using some Analytical Techniques

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Abstract*—A rapid, green, stability-indicating reversed-phase HPLC method was developed and validated for the simultaneous determination of benzbromarone and diclofenac, as well as the detection of related impurities and degradation products. The method addresses the need for robust quality control of high-volume pharmaceuticals, where cost pressures and evolving synthetic routes can introduce variability in product quality. Method development prioritised minimal environmental impact while ensuring selectivity and sensitivity suitable for routine release testing. Chromatographic separation was achieved on a Zorbax Eclipse Plus C18 (150 × 4.6 mm, 5 µm) column (Agilent). The method was challenged with forced degradation studies to demonstrate its stability-indicating capability and to confirm that degradation products do not interfere with the analyte peaks. Validation followed ICH-aligned criteria and included assessment of specificity, system suitability, forced degradation, limit of detection (LOD), limit of quantitation (LOQ), accuracy, and ruggedness. The retention time was recorded as Benzbromarone – 21.61 minutes, Diclofenac- 5.48 minutes, Benzbromarone Imp A- 10.17 minutes and Benzbromarone Imp C- 4.41 minutes.*

***Keywords*—** *Specificity, System Suitability, Force Degradation, LOQ, LOD and Precision.*

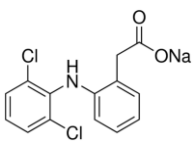
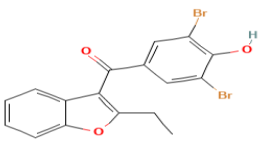
I. INTRODUCTION

The aim of the analytical method validation for associated compounds in Benzbromarone and Diclofenac Tablets 50 mg was to show that the process consistently yields dependable results that accurately reflect the product's quality attributes. The assay reported is a non-pharmacopoeial, in-house technique. Gout is treated with benzbromarone (3,5{-dibromo-4-hydroxyphenyl})[(2{-ethyl-1-benzofuran-3-yl}){methanone}], a uricosuric agent that is registered in several Asian, South American, and European countries [1]. Nonsteroidal anti-inflammatory drugs (NSAIDs), such as diclofenac, are commonly used to treat inflammation, fever, and discomfort in both human and veterinary medicine [2–3].

Benzbromarone and Diclofenac Tablets 50 mg analytical technique validation was conducted to demonstrate that the procedure consistently produces reliable findings that accurately reflect the product's quality features. The published assay is an internal, non-pharmacopoeial method. Some Asian, South American, and European countries have registered benzbromarone (3,5{-dibromo-4-hydroxyphenyl})[(2{-ethyl-1-benzofuran-3-yl}) methanone}] as a uricosuric agent for the treatment of gout [1]. Nonsteroidal anti-inflammatory drugs (NSAIDs), such as diclofenac, are commonly used to treat inflammation, fever, and discomfort in both human and veterinary medicine [4-5]. Citrus goods intended for human and animal consumption may accumulate benzbromarone [6] as a result of the compound's possible use in citrus plantations. Additionally, spraying these chemicals may cause them to leak into the soil and be absorbed by roots, contaminating the environment and perhaps harming both people and wildlife [7]. Therefore, before benzbromarone is widely used to regulate Huanglongbing (HLB), a sensitive and dependable extraction and quantification method for benzbromarone in citrus leaves, fruit peel, juice, roots, and soil is required [8]. While benzbromarone detection techniques have been documented for wastewater and a variety of animal and human tissues, there are currently no validated methods for the simultaneous analysis of benzbromarone in plant tissues[9].

The most used technique for identifying diclofenac in pharmaceutical dosage forms and biological materials is high-performance liquid chromatography (HPLC). In order to satisfy the needs for high accuracy and precision, cost-effectiveness, reproducibility, simplicity, and reliability, analytical techniques are constantly evolving [10-11]. The primary mechanism by which diclofenac reduces prostaglandin formation is via inhibiting cyclooxygenase enzymes. Additional pharmacological effects of diclofenac have been investigated by some researchers, but these results remain under investigation and should be regarded with caution [12-13].

Key words: Specificity, System Suitability, Force Degradation, LOQ, LOD and Precision

Structure		
Common Name	Diclofenac Sodium	Benzbromarone
IUPAC Name	2-[(2,6-Dichlorophenyl) amino] benzene acetic acid sodium salt	(3,5-dibromo-4-hydroxyphenyl)-(2-ethyl-1-benzofuran-3-yl)methanone
Empirical Formula	$C_{14}H_{10}Cl_2NNaO_2$	$C_{17}H_{12}Br_2O_3$
Molecular Weight	318.13	424.1

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II. MATERIALS AND METHODS

Material Name	Mfg. by	Purity (%)
Benzbromarone working standard	Aurobindo*	99.6
Diclofenac working standard	Glenmark*	99.0
Benzbromarone Impurity A	Synpure labs	98.60
Benzbromarone Impurity C	Simson	98.30

Chemicals and Reagents: Glacial Acetic Acid (AR), Methanol and Acetonitrile (HPLC Grade), Milli Q Water.
Equipment/Instrument: HPLC, Make Agilent and model 1260 infinity.
Chromatographic Conditions:

Instrument	:	HPLC with UV Detector and Thermostated sampler.
Column	:	Zorbax Eclips plus C18, 150 x 4.6 mm, 5µ
Flow rate	:	1.0 cm3 / min
Wavelength	:	231 nm
Injection volume	:	20 µL
Column Temperature	:	25°C
Sampler Temperature	:	5ºC
Run time	:	25 Minutes
Retention Time	:	Benzbromarone – 21.61 minutes: Diclofenac- 5.48 minutes: Benzbromarone Imp A- 10.17 minutes Benzbromarone Imp C- 4.41 minutes

Preparation of Mobile phase: Glacial Acetic acid: Acetonitrile: Water: Methanol (2.5:12.5:195:400).
Preparation of Diluent: The mobile phase is used as a diluent.
Preparation of standard solution: 20 mg of Benzbromarone, 20 cm^3 and add 5 cm^3 of Methanol, sonicate for 5 minutes to dissolve it. Made up the volume with mobile phase, Pipette out 2.0 cm^3 of the resulting solution in 20 cm^3. Further Pipette out 1.0 cm^3 of the resulting solution in 100 cm^3 make up volume with mobile phase (1 ppm).
Benzbromarone Impurity A Stock Solution: Accurately weigh and transfer 1 mg Benzbromarone Impurity A in a 10 cm^3 volumetric flask, add 5cm^3 of methanol volume with diluents. Standard stock solution (prepared above) (100 ppm).
Benzbromarone Impurity C Stock Solution: 1 mg Benzbromarone Impurity C in 5 cm^3 of methanol diluents. Standard stock solution (prepared above) (100 ppm).
System Suitability solution preparation: Transfer 1 cm^3 Benzbromarone stock solution and 1cm^3 of each Impurity A and C, and the volume with diluents. (1 ppm each concentration)
Placebo Preparation: Transfer the placebo powder eq. to 20 mg of Benzbromarone in 20 cm^3 and add 5 cm^3 of Methanol, sonicate for 5 minutes. Dilute with the mobile phase to the volume.
Sample preparation: Weighed accurately 50mg of Benzbromarone API into 50cm^3, 10cm^3 of methanol was added sonicate for 5 minutes. Dilute with the mobile phase to the volume. at 3000 rpm for 10minutes. Then the solution was a membrane filter (1000 ppm).
System suitability: 1) RSD for the area of the peak due to Benzbromarone in preparation should not be more than 5.0%. 2) Asymmetry for the peak due to Benzbromarone in six 2.0. 3) Theoretical plates for the peak due to Benzbromarone in preparation should be not less than 2000.

III. RESULTS AND DISCUSSION

Specificity: The parameters mentioned below are required to comply with during validation of the HPLC method for the assay test. Demonstrate the separation of the analyte from the placebo. Conduct the following forced degradation studies to obtain a degraded sample, preferably 10- 50% degradation and demonstrate the separation of the analyte from degradants. Specificity was assessed by evaluating the chromatograms of blank solution, placebo solution, standard solution and sample solution, i.e., 0.250 mg/mL and 0.475 mg/mL, respectively. For this purpose, 20 µl from the placebo solution, standard solution and sample solution were injected into the HPLC system separately, and the chromatogram results [14].
System suitability: The equipment (system) is working correctly, under highly precise conditions. The autosampler is taking precisely the same volume each time, or if your manual injection is okay, etc. Hope this was helpful. System suitability testing is an integral part of many procedures above-mentioned system suitability parameters are not a must.

TABLE 1: System suitability (Benzbromarone Working Standard):

Injection No.	Area	Asymmetry	Theoretical Plates
1	859955	1.03	11893
2	813113	1.07	12339
3	806293	1.02	12178
4	818163	1.04	12164
5	823892	1.03	12168
6	834344	0.99	12175
Mean	**825960**	**1.03**	**12153**
Std. Dev.	**19198.22**	**-**	**-**
% RSD	**2.32**	**-**	**-**

Peak Purity Table

Sample Name	Retention Time	Peak Purity
Blank preparation	NA	NA
Placebo preparation	NA	NA
Benzbromarone	21.56	1.000
Benzbromarone Impurity C	4.43	1.000
Benzbromarone Impurity A	10.23	1.000

Forced degradation: Forced degradation, commonly known as stress testing, is carried out to demonstrate the specificity to develop a stability-indicating analytical method, using high-performance liquid chromatography (HPLC), i.e., a single analytical method that is capable of separating the degrading peaks from the drug substance. Thermal degradation and Photolytic degradation. To illustrate the stability-indicating power of analytical techniques, forced-degradation experiments, or stress testing, of a pharmacological substance are used [15-17]. While regulatory guidance materials stress the necessity of these investigations, they do not specify the pH, temperature ranges oxidising agents to employ, or the circumstances under which to use. The form of stress testing will vary on the individual drug substance, according to International Conference on Harmonisation (ICH) Q1A (R2) (1). As a result, forced-degradation study designs differ from

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one firm to the next, with common experimental designs focusing on the four primary pharmaceutically important degradation mechanisms: thermolytic, hydrolytic, oxidative, and photolytic [18-19].

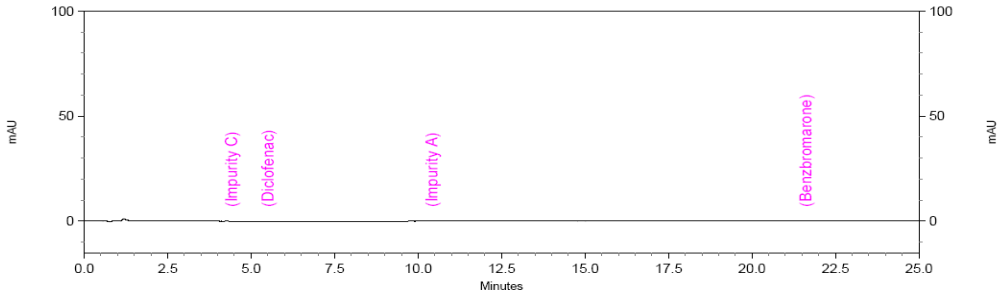

Figure 1: Placebo solution chromatogram in specificity

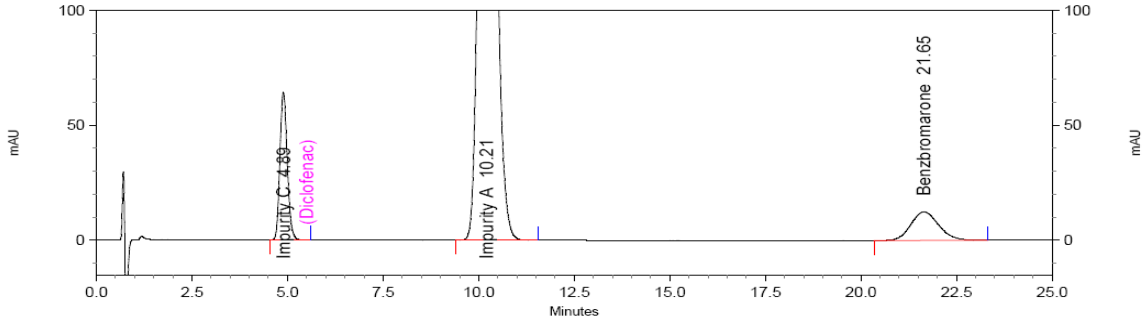

Figure 2: Benzbromarone Impurity-A

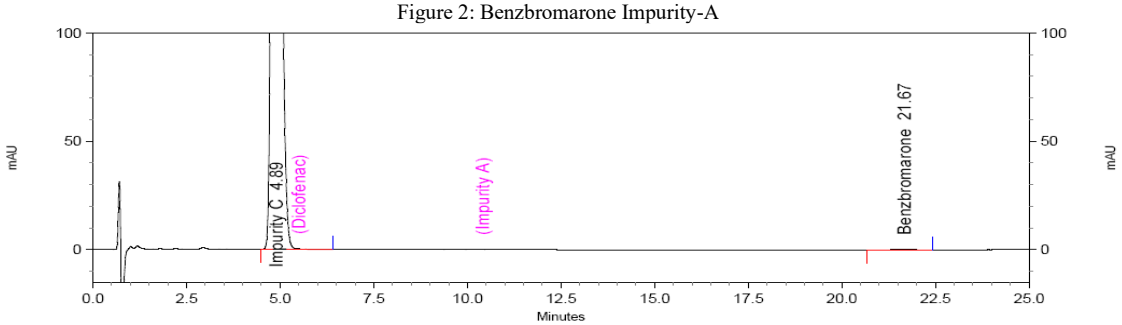

Figure 3: Benzbromarone Impurity-C

TABLE 2: System suitability (Benzbromarone Working Standard)

Injection No.	Area	Asymmetry	Theoretical Plates
1	846504	1.00	9618
2	849509	0.98	9447
3	851613	0.97	9685
4	838128	1.00	9636
5	820727	0.96	9567
6	836069	1.00	9649
Mean	**840425**	**0.99**	**9600**
Std. Dev.	**11462.17**	**-**	**-**
% RSD	**1.36**	**-**	**-**

The forced-decomposition settings were chosen based on data from accelerated pharmacological research and a thorough grasp of the product's decomposition mechanism. Forced degradation investigations are clearly stipulated by the International Conference on Harmonisation (ICH) recommendations. These studies provide data that can be used to help detect probable degradants. It also shows how pharmaceutically active compounds are degraded. Forced degradation experiments can be used to assess the intrinsic stability of a pharmacological molecule. Forced degradation experiments can also be used to assess possible polymorphism or enantiomeric compounds, as well as variations in drug-related degradation and excipient interferences.

TABLE 3: Peak Purity Table for Benzbromarone

Name of Sample	**Area**	**Peak Purity**	**% Degradation**
Blank Preparation (Untreated)	Not detected	NA	---
Placebo Preparation (Untreated)	Not detected	NA	---
API Preparation (Untreated)	404819123	1.000	---
API mixture Preparation (Untreated)	404685904	1.000	---
Test Preparation (Untreated)	458474033	1.000	---
Placebo Preparation (Thermal Degradation)	Not detected	NA	---
API Preparation (Thermal Degradation)	406677440	1.000	0.46

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API Mixture Preparation (Thermal Degradation)	405697206	1.000	0.25
Test Preparation (Thermal Degradation)	367621131	1.000	19.74
Placebo Preparation (Photolytic Degradation)	Not detected	NA	---
API preparation (Photolytic Degradation)	405339677	1.000	0.13
API mixture preparation (Photolytic Degradation)	407336702	1.000	0.65
Test Preparation (Photolytic Degradation)	397680072	1.000	13.21

Benzbromarone: Base degradation, Acid degradation and Oxidative degradation.

TABLE 4: System suitability (Benzbromarone Working Standard)

Sr. No. Injection No.	Area	Asymmetry	Theoretical Plates
1	844485	0.96	7755
2	841502	0.96	7731
3	870198	0.99	7723
4	842609	0.97	7704
5	845798	0.94	7768
6	830908	0.94	7669
Mean	**845917**	**0.96**	**7725**
Std. Dev.	**13017.96**	-	-
% RSD	**1.54**	-	-

TABLE 5: Peak Purity Table for Benzbromarone

Name of Sample	*Area*	Peak Purity	% Degradation
Blank Preparation (Base Degradation)	Not detected	NA	---
Placebo Preparation (Base Degradation)	Not detected	NA	---
API Preparation (Base Degradation)	403747363	0.996	0.27
API mixture Preparation (Base Degradation)	386678267	0.995	4.43
Test Preparation (Base Degradation)	337890794	0.991	26.20
Blank Preparation (Acid Degradation)	Not detected	NA	---
Placebo Preparation (Acid Degradation)	Not detected	NA	---
API Preparation (Acid Degradation)	399189293	0.998	1.39
API mixture Preparation (Acid Degradation)	399425710	0.995	1.30
Test Preparation (Acid Degradation)	381380208	0.991	16.75
Blank Preparation (Oxidative Degradation)	Not detected	NA	---
Placebo Preparation (Oxidative Degradation)	Not detected	NA	---
API Preparation (Oxidative Degradation)	404230342	0.986	0.15
API mixture Preparation(Oxidative Degradation)	411580665	0.972	1.70
Test Preparation (Oxidative Degradation)	397253131	0.985	13.30

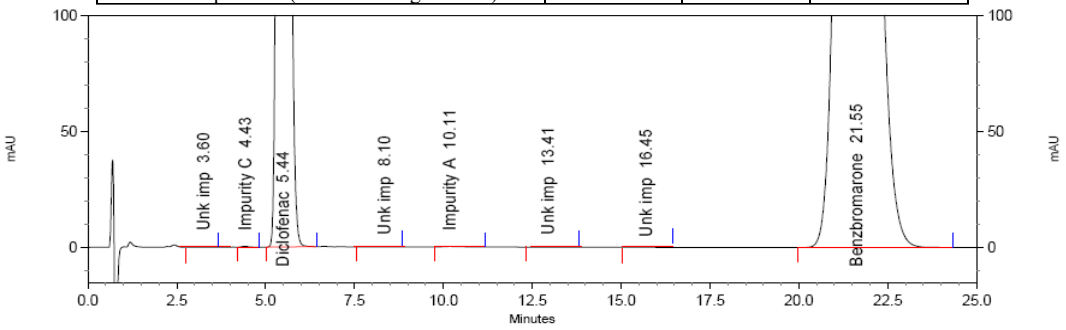

Figure 4: Acid degraded sample solution chromatogram in Force degradation

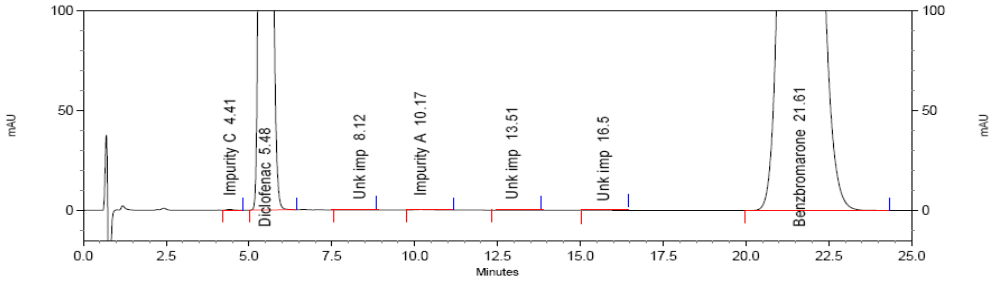

Figure 5: Base-degraded sample solution chromatogram in Force degradation

Acceptance Criteria: 1. The Benzbromarone Peak, 2. Peak purity for Benzbromarone peak in all degraded sample to or more than 0.950.

Limit of Quantisation (LOQ) and LOD: The lowest analyte concentration that can be quantitatively identified with a defined accuracy and precision is known as the Limit of Quantification (LOQ). The determination of LOQ, on the

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other hand, is based on predetermined acceptance criteria and performance. Blank and standard solution prepared as per the given specificity parameter. From the above solution LOQ solution was prepared as given in the table below. The system suitability obtained is given in the following table.

TABLE 6: System suitability (Benzbromarone Working Standard)

Injection No.	Area	Asymmetry	Theoretical Plates
1	885871	1.08	11388
2	852240	1.03	11781
3	840457	1.06	11779
4	865780	1.06	11578
5	875796	1.02	11444
6	848514	1.02	11835
Mean	**861443**	**1.05**	**11634**
Std. Dev.	**17400.07**	**-**	**-**
% RSD	**2.02**	**-**	**-**

Limit of Quantitation was solution was prepared as follows.

TABLE 7: Precision at LOQ Level

Sr. No.	Benzbromarone	Benzbromarone Impurity A	Benzbromarone Impurity C
1	61169	78675	48559
2	58547	87248	47094
3	61848	92271	44716
4	59246	96204	49160
5	59859	82212	45488
6	61154	79627	48167
Mean	**60304**	**86040**	**47197**
Std. Dev.	**1285.23**	**7122.01**	**1774.03**
% RSD	**2.13**	**8.28**	**3.76**

TABLE 8: LOQ

Name	LOQ in % w.r.t. test concentration	S/N Ratio
Benzbromarone	0.002	19.3
Benzbromarone Impurity A	0.048	12.0
Benzbromarone Impurity C	0.021	11.3

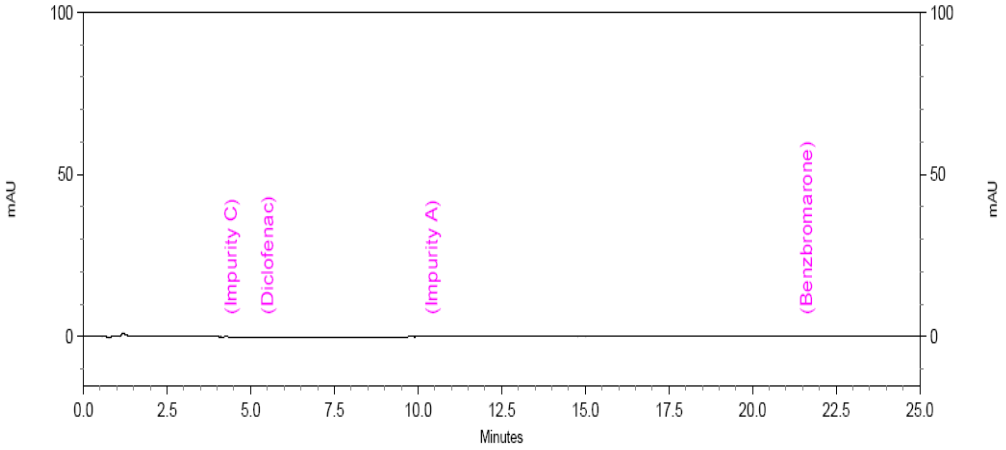

Figure-6: Blank solution chromatogram in LOD/LOQ Parameter

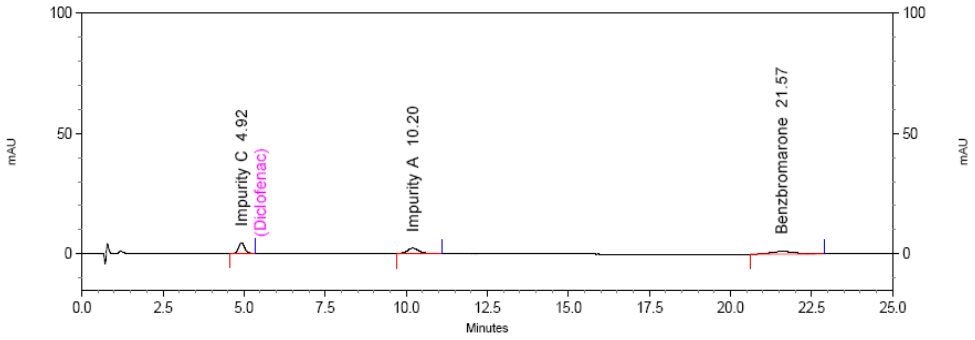

Figure 7: LOQ solution chromatogram

Acceptance Criteria: RSD for the area of impurity should not be more than 10.0%. Report the value of Quantitation limit.

TABLE 9: Area at LOD Level

Sr. No.	Benzbromarone	Benzbromarone Impurity A	Benzbromarone Impurity C
1	22078	24818	18096
2	19878	26639	15648
3	18033	23728	17682
Mean	19996	25062	17142

TABLE 10: LOD:

Name	LOD in % w.r.t. test concentration	S/N Ratio
Benzbromarone	0.001	3.4
Benzbromarone Impurity A	0.016	3.4
Benzbromarone Impurity C	0.007	3.9

Linearity and Range: Linearity is assessed by infusing a series of stock/diluted stock standards into the solvent/mobile phase at a minimum of five different concentrations over a design

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working range of 50% and 150 %. The ability of an analytical procedure to produce control results, either immediately or with the aid of well-defined variables, is known as linearity. The interval between the upper and lower value, which is determined with precision, accuracy and linearity using the set technique, is the range of the analytical method. This is the concentration range over which the linearity test will be performed.

TABLE 11: System suitability (Benzbromarone Working Standard)

Injection No.	Area	Asymmetry	Theoretical Plates
1	878528	1.06	10714
2	868209	1.03	10481
3	879795	1.03	10767
4	874809	1.03	10761
5	867280	1.09	10878
6	887393	1.01	10721
Mean	**876002**	**1.04**	**10720**
Std. Dev.	**7599.34**	**-**	**-**
% RSD	**0.87**	**-**	**-**

TABLE 12: Linearity of Benzbromarone

Linearity Level	LOQ Level	10% Level	20% Level	50% Level	80% Level	100% Level	120% Level	150% Level	200% Level
Conc. (ppm)	0.048	1.025	2.051	5.127	8.204	10.254	12.305	15.382	20.509
Inj.1	61169	1246860	2595783	6559503	10740966	12948099	16359382	18537968	25420106
Inj.2	58547	1253269	2603714	6552098	10708610	12990541	16467065	19431733	25447098
Inj.3	61848	1248097	2589210	6549544	10669195	12970687	16423241	19221314	25353295
Inj.4	59246	1256391	---	---	---	---	---	---	25362176
Inj.5	59859	1247008	---	---	---	---	---	---	25358684
Inj.6	61154	1246254	---	---	---	---	---	---	25365383
Mean	60304	1249647	2596236	6553715	10706257	12969776	16416563	19063672	25384457
Std. Dev.	1285.2	4177.25	7262.59	5172.66	35943.31	21235.67	54151.24	467271.1	39218.17
% RSD	2.13	0.33	0.28	0.08	0.34	0.16	0.33	2.45	0.15

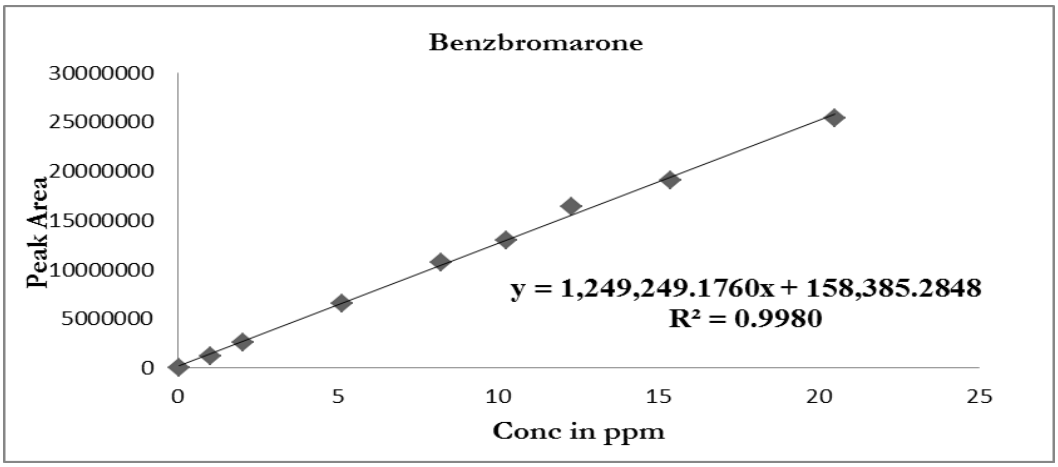

Figure 8: Linearity Benzbromarone

TABLE 13: Linearity Benzbromarone Impurity A

Linearity Level	LOQ Level	10% Level	20% Level	50% Level	80% Level	100% Level	120% Level	150% Level	200% Level
Conc. (ppm)	0.241	0.258	0.516	1.290	2.064	2.580	3.096	3.870	5.160
Inj.1	78675	52510	147128	345777	560029	693631	856413	1023821	1409203
Inj.2	87248	52196	150628	350248	549835	686222	870134	1073212	1414139
Inj.3	92271	49943	153782	364030	570410	697063	868256	1056852	1402149
Inj.4	96204	48050	---	---	---	---	---	---	1411495
Inj.5	82212	49349	---	---	---	---	---	---	1412328
Inj.6	79627	50785	---	---	---	---	---	---	1394538
Mean	**86040**	**50472**	**150513**	**353352**	**560091**	**692305**	**864934**	**1051295**	**1407309**
Std. Dev.	**7122.01**	**1710.59**	**3328.50**	**9514.07**	**10287.6**	**5540.75**	**7439.19**	**25160.0**	**7518.95**
% RSD	**8.28**	**3.39**	**2.21**	**2.69**	**1.84**	**0.80**	**0.86**	**2.39**	**0.53**

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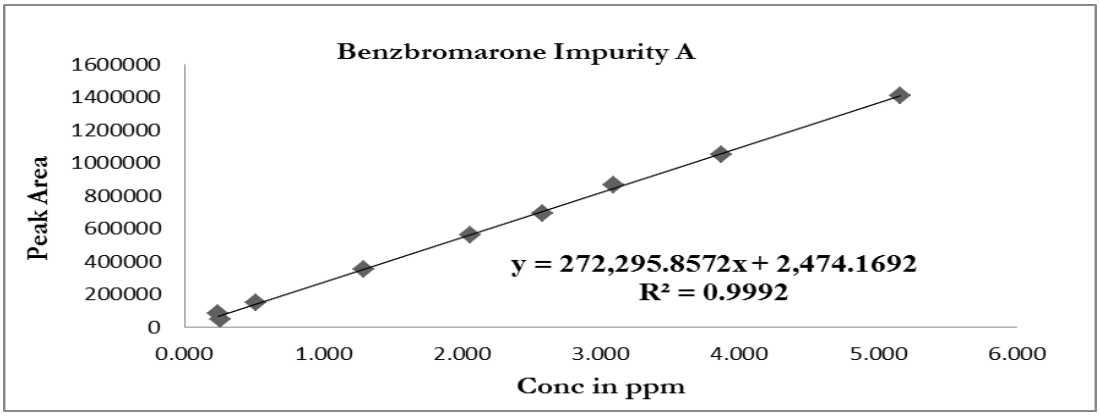

Figure 9: Linearity Benzbromarone Impurity A

TABLE 14: Linearity Benzbromarone Impurity C

Linearity Level	LOQ Level	10% Level	20% Level	50% Level	80% Level	100% Level	120% Level	150% Level	200% Level
Conc. (ppm)	0.105	0.102	0.205	0.512	0.820	1.025	1.230	1.537	2.049
Inj.1	48559	90095	168477	412510	689105	866924	1064093	1263990	1749407
Inj.2	47094	81760	169624	421411	683694	848679	1063822	1318294	1740375
Inj.3	44716	79834	174494	436566	686613	848053	1053847	1296205	1734033
Inj.4	49160	83615	---	---	---	---	---	---	1733834
Inj.5	45488	86966	---	---	---	---	---	---	1719988
Inj.6	48167	87210	---	---	---	---	---	---	1765404
Mean	**47197**	**84913**	**170865**	**423496**	**686471**	**854552**	**1060587**	**1292830**	**1740507**
Std. Dev.	**1774.03**	**3841.00**	**3194.70**	**12162.7**	**2708.31**	**10719.0**	**5838.87**	**27308.9**	**15534.2**
% RSD	**3.76**	**4.52**	**1.87**	**2.87**	**0.39**	**1.25**	**0.55**	**2.11**	**0.89**

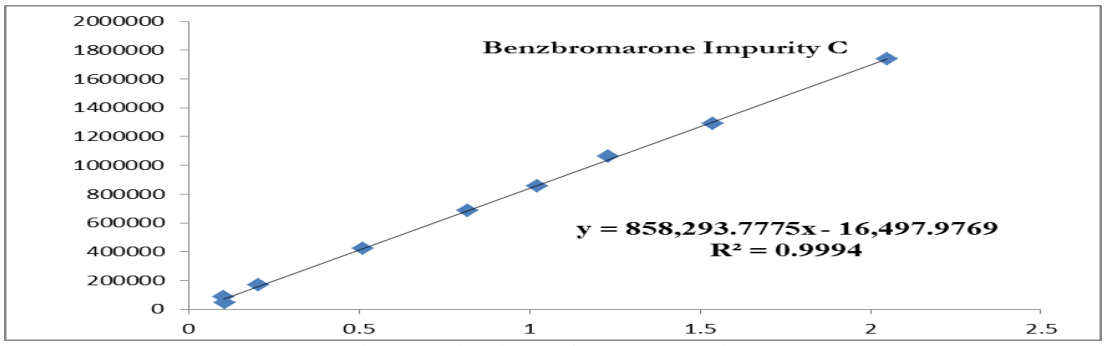

Figure-10: Linearity Benzbromarone Impurity C

TABLE 15: Coefficient of Correlation

Component Name	Coefficient of correlation	Criteria
Benzbromarone	0.9980	Not less than 0.99
Benzbromarone Impurity A	0.9992	Not less than 0.99
Benzbromarone Impurity C	0.9994	Not less than 0.99

TABLE 16: Slope of regression line, y-Intercept and residual sum of squares

Component Name	Slope of the regression line	y-Intercept	Residual sum of square
Benzbromarone	1249249.1760	158385.2848	1202821894490.4
Benzbromarone Impurity A	272295.8572	2474.1692	1456867802.93
Benzbromarone Impurity C	-16857444.0237	-16474.2781	1651196676.33

TABLE 17: Response Factor

Component Name	Relative Retention Time	Response Factor
Benzbromarone	0.25	1.04
Benzbromarone Impurity A	0.36	3.10
Benzbromarone Impurity C	1.13	0.62

Intermediate Precision (Ruggedness):

The robustness/ruggedness of an analytical procedure is a measure of its ability to remain unaffected by tiny, but deliberate modifications in method parameters and indicates its reliability during routine use, according to the definition [20].

Ruggedness is usually defined as the lack of influence of operational and environmental variables of the analytical procedure on the test results. The goal of a ruggedness test is

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to identify the elements that have a significant impact on measurement findings and to determine how closely these factors must be controlled. Ruggedness tests do not identify the best circumstances for a particular test procedure.

TABLE 18: System suitability (Benzbromarone Working Standard)

Injection No.	Area	Asymmetry	Theoretical Plates
1	842505	1.02	10575
2	849245	1.09	10354
3	843286	1.05	10567
4	845604	1.10	10829
5	849694	1.04	10770
6	849163	1.04	10785
Mean	**846583**	**1.06**	**10647**
Std. Dev.	**3221.17**	**-**	**-**
% RSD	**0.38**	**-**	**-**

TABLE 19: % of Impurities

Serial No.	Benzbromarone Related compound C	Benzbromarone Related compound A	Unknown Individual Max Impurity	Total Impurities
Test preparation 1	0.363	0.225	0.057	0.530
Test -2	0.362	0.229	0.056	0.548
Test -3	0.364	0.230	0.056	0.552
Test-4	0.366	0.223	0.055	0.539
Test-5	0.367	0.225	0.059	0.553
Test - 6	0.372	0.227	0.054	0.553
Mean	0.366	0.227	0.056	0.546
Minimum	0.362	0.223	0.054	0.530
Maximum	0.372	0.230	0.059	0.553
Difference	**0.010**	**0.007**	**0.005**	**0.023**

Acceptance Criteria:

a) Individual known impurities should not be more than 0.5%.
b) Individual Benzbromarone Related Compound C impurity should not be more than 2.0%.
c) Individual Benzbromarone Related Compound A impurity should not be more than 2.0%.
d) The individual unknown impurity should not be more than 0.2%.
e) Total impurities should not be more than 2.0%
f) Difference between the lowest value and the highest value of individual impurities 0.03% absolute.
g) Difference between the lowest value and the highest value of total impurities 0.05% absolute.
h) The difference between individual impurities and total impurities of twelve results (six each of) should not be more than 0.05% absolute.

IV. CONCLUSION

The observations and results for all validation parameters—specificity, limit of quantitation (LOQ, recovery), solution stability, filter compatibility, and system suitability—fell well within the predefined acceptance criteria. These conclusions were supported by analyses of blank, placebo, diluted standard, individual known-impurity, test, and test-spiked preparations. Blank, placebo, and individual known-impurity preparations showed no detectable benzbromarone peak. Linearity, precision, and accuracy data demonstrated the method is precise and accurate across the validated range from LOQ to 200% of the working concentration. Ruggedness was confirmed under intermediate-precision conditions, with results remaining within acceptable limits. Stability studies indicated that the standard preparation remained stable for up to 24 hours and the test preparation for up to 16 hours when stored at 5 °C. System suitability parameters met the acceptance criteria established during validation. Because all results satisfy the acceptance criteria, the method is considered validated and suitable for routine quality control and stability testing.

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