ERDOSTEINE CAPSULES - MONOGRAPH.

HPLC ASSAY AND RELATED SUBSTANCE

Column	Eurospher 100, C18, 25 x 0.40 cm 5µ
Mobile Phase	Buffer pH 2.0*: Acetonitrile (88:12 v/v)
* Buffer pH 2	Potassium dihydrogen phosphate (KH_2PO_4) - 0.68g
	Hepatane sulphonic acid - 1.01g
	Phosphoric acid (85%) - 4.6mL & Water - to 1000mL adjust pH to 2.0 with Sodium hydroxide (35% w/v)
Flow rate	1.5mL/min
Sample volume	10µL
Detector	UV at 220nm, AUFS 0.01

Mobile phase proportions and flow rate may be varied in order to achieve the required system suitability

ALL SOLVENTS USED MUST BE HPLC GRADE

ALL SOLUTIONS MUST BE FRESH DAILY

STANDARD PREPARATION

Accurately weigh about 14mg Erdosteine A.S. into a 50mL volumetric flask. Add about 35mL of mobile phase and sonicate to dissolve. Make up to volume with mobile phase. This is the Standard solution.

SYSTEM SUITABILITY SOLUTION

Weigh about 6mg of Metabolite 1 into a 20mL volumetric flask. Dissolve in and make up to volume with Standard solution.

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SYSTEM SUITABILITY TEST

Inject the System Suitability Solution.

The retention time of the Erdosteine peak is about 6 minutes and Metabolite 1 peak is about 7.5 minutes. The resolution factor between these two peaks (calculated according to USP) should be not less than 2.5.

The tailing factor of the Erdosteine peak (calculated according to USP) should be not greater than 1.5.

The relative standard deviation, calculated for 5 replicate injections of the standard preparation must be not more than 2.0%.

SAMPLE PREPARATION

Weigh 20 capsules. Transfer as completely as possible the contents of the capsules into a suitable tarred container and determine the average content weight per capsule.

Mix the combined contents and accurately weigh about 60mg of the powder into a 200mL volumetric flask.

Add 150mL of mobile phase and sonicate for 15 minutes. Make up to volume with mobile phase. Filter through a 0.45µ membrane filter.

PROCEDURE

Inject the Standard and Sample solutions into the chromatograph and determine the peak area of Erdosteine in each chromatogram using a suitable integrator.

CALCULATIONS

$$\frac{\text{Pk area smp x Std wt * (mg) x Avg cap cont wt(mg) x 400}}{\text{Pk area std x Smp wt(mg) x Dose(mg / cap)}} = \%\ \text{Erdosteine of labeled claim}$$

* Std wt is corrected in accordance with % Assay and % Water.

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CONTENT OF METABOLITE 1

During the HPLC determination of Erdosteine in capsules, the evaluation of Metabolite 1 can be done from the same chromatogram.

$$\frac{\text{Pk area Met 1}}{\text{Pk area Erdosteine}} \times F^{*} \times 100 = \% \text{ of Metabolite 1}$$

* F = 4(response factor of Metabolite 1 relative to Erdosteine).

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TYPICAL GRAPH

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SYSTEM SUITABILITY GRAPH

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DISSOLUTION

FOR GENERAL PROCEDURE FOR ALIGNMENT OF EQUIPMENT & WITHDRAWAL OF SAMPLES REFER TO USP 23, SUPPLEMENTS & RELEVANT SOP's

DISSOLUTION TEST PROCEDURE

Equipment	:USP 6-vessel dissolution Apparatus 2 (Paddle)
Medium	:0.1M HCl
Volume	:1000mL
Temperature	:37.0°C ± 0.5°C
Stirring rate	:100 rpm

Note: ENSURE THAT THE SPIRALS ARE NOT TIGHT

SAMPLE PREPARATION

Unless otherwise specified 10mL samples are withdrawn from each vessel after 30 minutes and filtered through filter tips (Filter reagent CPO7H245, Cat. No. 178-3985-01 Technicon).

Pipet 2.5mL of above solution into a 25mL volumetric flask and make up to volume with dissolution medium (this is the Sample Solution).

STANDARD PREPARATION

Accurately weigh about 15mg Erdosteine A.S. into a 50mL volumetric flask. Add 30mL of dissolution medium and sonicate for 15 minutes.

Make up to volume with dissolution medium. Pipet 2.5mL of above solution into a 25mL volumetric flask and make up to volume with dissolution medium.

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ANALYTICAL PROCEDURE

Measure the absorbance of the Standard and Sample solutions at the maximum wavelength at about 236nm using dissolution medium as blank.

CALCULATION

$$\frac{\text{Abs smp x Conc std * (mg/mL) x Diss vol(mL) x 100}}{\text{Abs std x Dose(mg/caps)}} = \text{\% Erdosteine dissolved}$$

* Take into account the % Water and the % Assay of the Erdosteine standard.

ACCEPTANCE CRITERIA

Not less than 75% (Q) after 30 minutes.

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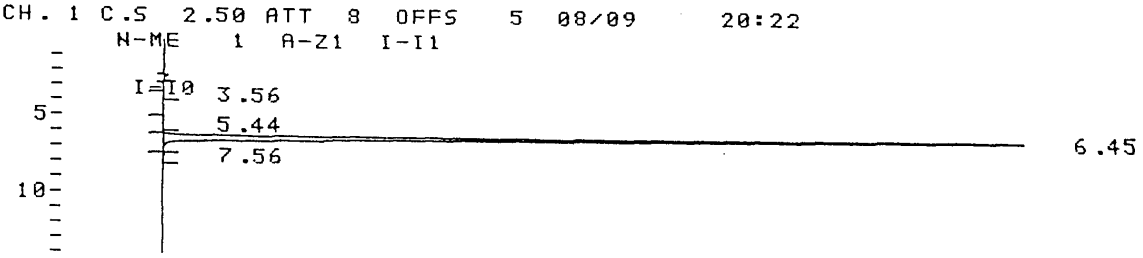
TYPICAL GRAPH



ERDOSTEINE ASSAY

Erdosteine Caps

SAMPLE: Erdosteine Capsules IAG-792

D-2500 08/09/9 20:22

METHOD: EN TAG: 12 CH: 1 VIAL: 16

FILE: 7 CALC-METHOD: AREA% TABLE: 0 CONC: AREA

NO.	RT	AREA	CONC	HEIGHT	BC
1	3.56	742	0.035	88	BB
2	5.44	4080	0.195	145	BB
3	6.45	2083062	99.538	215107	BB
4	7.56	4856	0.232	440	BB
TOTAL		2092740	100.000	215780	