Japanese Pharmacopoeia, 18th Edition Analysis of Saccharin Sodium Hydrate - 2.00 Change of Conditions Specified within General Theory of Chromatography

Adjustable range of chromatography conditions has been specified in [2.00 General theory of chromatography] which has been stipulated based on the contents harmonized and agreed upon by Japanese/U.S./European Trilateral Pharmacopoeia at the first supplemental public comment of Japanese Pharmacopoeia, 18th Edition, which was disclosed in September 2021. Based on this, the acceptable changeable range of conditions is widened, enabling easier requirement study.

In this report, analysis was implemented by changing the column size within the range specified in [2.00 General theory of chromatography] for JP18 Purity test (5) of saccharin sodium hydrate.

The change of analytical conditions is for the content to be applied from the first supplemental revision of JP18 and is not for the content announced in the past. Accordingly, this Technical Note is simply just for reference.

Changeable Items in JP

In [2.00 General theory of chromatography], the change of LC columns and instrumental conditions is allowed by satisfying the requirements of system suitability.

Items in Pharmacopoeia		Japanese Pharmacopoeia <Public Comment Proposal in September 2021> International Harmonization by Trilateral Pharmacopoeia
Stationary phase	Particle size	Can be reduced down to a maximum of 50%, but cannot be increased (packed column)
	Film thickness	-50% ~ +100%(Capillary column)
Column size	**Length**	**-70% ~ +100%**
	Inner diameter	±50%
Instrument	Column temperature	±10%
	Temperature program	Adjustment of temperature is acceptable as described above. Adjustment of temperature increase rate and retention time for each temperature is tolerable within ±20%.
Carrier gas	Flow rate	±50%
	Injection volume and Split ratio	If the requirement of system suitability is within the established tolerable range, injection volume and split ratio can be changed. For reducing the injection volume or increasing the split ratio, it is necessary to pay particular attention to detection (detection limit) of peak response and reproducibility. Increase of injection volume or decrease of split ratio is allowed only if linearity and resolution of measurable peak satisfy the requirement of system suitability.
	Injection port temperature and conditions for transfer line temperature at static head space	±10℃ unless decomposition or concentration occurs

JP18 Purity Test (5) of Saccharin Sodium Hydrate

Referring to JP18 Purity Test of saccharin sodium hydrate as an analysis example, analysis was implemented by changing the column size within the changeable range specified in JP [2.00 General theory of chromatography]

【Purity Test (5)】

＜Test conditions＞

Detector: Hydrogen flame ionization detector

Column: Diatomaceous earth for gas chromatography of 180 – 250 µm coated with succinic acid diethylene glycol polyester for gas chromatography at a ratio of 3% is packed in the tube of 3 mm in inner diameter, and 1 m in length.

Column temperature: Constant temperature in the vicinity of 200℃

Injection port temperature: Constant temperature in the vicinity of 225℃

Detector temperature: Constant temperature in the vicinity of 250℃

Carrier gas: Nitrogen

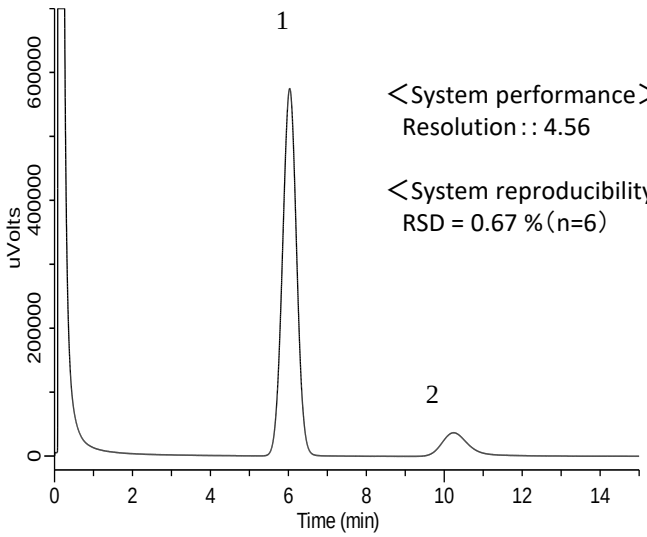
Floe rate: **Adjust so that the retention time of caffeine may become approximately 6 min.**

＜System suitability＞

System performance: When operated in the above conditions, for each 1 µL of standard solution, internal standard substance and o-toluensulfonamide need to be eluted in order and separation degree needs to be higher than 2.0.

System reproducibility: When test was repeated 6 times in the above conditions, for each 1 µL of standard solution, the relative standard deviation of the ratio of peak height of o-toluensulfonamide against peak height of internal standard substance needs to be less than 2.0%.

Column size described in test method is 1 m and acceptable changeable range is -70% - +100%. Since this corresponds to 0.3 m ≦ 1 m ≦ 2.0 m, test was implemented by changing the column length to 0.5 m for this analysis.



＜System performance＞
Resolution: : 4.56

＜System reproducibility＞
RSD = 0.67 %（n=6）

1. Caffeine (I.S.)
2. *o*-toluensulfonamide

(Standard solution)

Conditions

Column	: DEGS 3% Uniport HP 60/80 Glass 1/4” O.D. x 0.5 m x 3.0 mmI.D.
Col.Temp.	: 200 ℃
Carrier Gas	: N_2 32 mL/min
Detector	: FID 250℃
Injection	: 225℃
Sample Size	: 1 µL

Column pressure: 35 kPa

Retention time of caffeine: 6.03 min

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