阴离子表面活性剂滴定法测定盐酸氯派丁片的含量和含量均匀度

李　伟(徐州 221002 徐州市药品检验所)

摘要　目的:建立盐酸氯哌丁片的含量和含量均匀度测定方法。方法:采用阴离子表面活性剂滴定法测定盐酸氯哌丁片的含量和含量均匀度。以磺基丁二酸钠二辛酯试液为滴定剂,二甲基黄—溶剂蓝 19 混合液为指示剂,氯仿为萃取剂。结果:平均回收率为 99.8 %±0.58 %。结论:方法简便省时,结果准确。

关键词　盐酸氯哌丁;含量均匀度;阴离子表面活性剂;滴定

Cloperastine Hydrochloride tablets is Antitussive, used in the treatment of unproductive cough. There were some reports for the determination of cloperastine hydrochloride, such as non-aqueous titration[1], According to the chemical properties of cloperastine hydrochloride, a method by titration with an anionic surfactant[2] was developed.

Experimental

Reagents

Cloperastine Hydrochloride (Liaoyuan pharmaceutical Co, Ltd. Jilin. The purity of it was 99.62 %), cloperastine hydrochloride tablets (Liaoyuan pharmaceutical Co Ltd Jilin, Batch No 970701, 970703; Zhenjiang No.2 pharmaceutical factory, Batch No 960401-1). Sodium dioctylfosuccimate TS, dimethyl yellow-solvent blue 19 IS, sulfuric acid (AR), perchloric acid(AR).

Linearity

Standard preparation of reference solution: Dissolve about 0.25g of cloperatus hydrochloride, accurately weighed in 50 ml of water in a volumaetric flask.

1.0, 1.5, 2.0, 2.5, 2.75, 3.0 ml of cloperatus hydrochloride reference solution were transferred respectively to 100 ml iodine flasks, add 4, 3.5, 3, 2.5, 2.25, 2.0 ml of water, 20 ml of chloroform and 5 ml of dilute sulfuric acid was added. Then 1 ml of dimethyl yellow-solvent blue 19 IS was added. The sample was titrated with sodium dioctylsulfosuccimate TS. Shake vigrously at near end point and contituely titrate until the chloroform layer changes from green to reddish-grey. Volume of sodium dioctylsulfosuccimate was 3.60, 5.38, 7.21, 8.91, 9.96, 10.93 ml respectively. The regression equations of volume of sodium dioctylsulfosuccimate TS against amounts(mg) was C(mg) = 1.3715 V + 0.1118, with a correlation coefficient r = 0.9997. The calibration curve showed a good linearily over the range tested.

Assay

Weigh accurately and powder 10 tablets. A quantity of powdered tablets, which in equivalent to 10 mg of cloparastine hydochloride was weighed accurately and patinto 100 ml iodine flask, add 5 ml of water, carry out the procedure in as described Linearity, beginning at the words"add 20 ml of chloroform..." . perfrom a reference titration using 10 mg of cloparastine hydrochloride CRS. Calculate the content of $C_{20}H_{24}ClNO \cdot HCl$ from the titration.

Tab1　Samples assay resules (labeled %, $n = 3$)

Lot No	method Ⅰ	method Ⅱ
960401-1	95.7	95.2
970701	96.9	97.5
970703	99.6	100.6

Ⅰ this method Ⅱ ministry method

Uniformity

Transfer 1 tablet with 5 ml of water and 20 ml of chloroform, previously crushed of finely powdered, to 100 ml iodine flask, Carry out the procedure described in Linearity, beginning at the words "add 5 ml of dilute sulfuric acid..." . Perform a reference titration using 2 ml of stock solution. Calculate the content of each tablet, comly with the content uniformity for tablets (Appendix XE, CP2000, Vol. 2) .

Precision test

Weigh accurately and powder 10 tablets. Weigh accurately a quantity of powdered tablets, equivalent to 10 mg of cloparastine hydrochloride in 100 ml iodine flask. Carry out the assay as described, beginning at the words "add 5 ml of water..." , calculate the RSD was 0.46 %($n=5$) .

Tab2 Results of Determination of the content uniformity

Lot No	Content %	$\bar{x}$	$A+1.80s$
960401-1	87.5 ~ 100.8	95.4	12.6
970701	94.1 ~ 102.2	98.64	5.6
970703	85.8 ~ 106.8	98.16	14.2

Recovery test

Weighed accurately cloperatus hydrochloride about 5 ~ 15 mg, than supplement material was added according to ratio case of prescription. Carry out the assay as described, beginning at the words "add 5 ml of water..." . Perform a reference titration using 2 ml of the test stock solution and calculate the content of cloparatus hydrocloride from the titration.

The ranges of recovery for 5.1 mg to 14.8 mg of cloperatus hydrochloride were from 99.2 % to 100.8 %, and the average recovery was 99.8 %. The relative standard deviation in all applied analysis was less than 0.58 %($n=6$) .

Discussion

The method developed in this paper is simple, fast, accurate and sensitive. weight variation of tablets need not to be tested again after coating. The content uniformity must be tested.

References

1 Drugs Specifications Promulgated by the Ministry of Public Health, PR China WS_1-30-82-89:117.

2 AnDK, Zhangzx, shangls. pharmaceutical Analysis. Jinan: Jinan Press, 1992:1183.

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