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ASSAY or CALAMINE LOTION(การวิเคราะห์ CALAMINE LOTION)

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ORIGINAL ARTICLE

ASSAY OF CALAMINE LOTION

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ABSTRACT

Acid-base titration for total zinc oxide content in calamine lotion with pink or red colour using bromocresol purple as indicator is simple, rapid and having high accuracy and precision. End point detection is sharp and this method is more accurate than complexometric titration. (Th. J. Pharm. Sci., Vol. 12 No. 4, 309-314 (1987))

Keywords :

Acid-base titration, calamine lotion, bromocresol purple.

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INTRODUCTION

In the determination of calamine and zinc oxide, in term of total zinc oxide content in calamine lotion, acid-base titration (1) can not be carried out due to the interference from red colour of Poncoau 4R, erythrocine or camoisine to the end point detection when methyl orange is used as indicator.

To solve this problem, an appropriated indicator was selected based on the usefullness of interfering colour to enhance sharpness in end point detection. Complexometric titrations were also performed in order to compare with that of the selected method.

EXPERIMENTAL

Materials

Chemicals : Calamine B.P. 1973 (Durham Chemical Ltd., England); zinc oxide U.S.P.XIX, bentonite U.S.P.XIX, menthol U.S.P.XIX (China National Chemical Imp. & Exp. Corp., China) ; sulfuric acid AR, sodium hydroxide AR, hydrochloric acid AR, triethanolamine Lab. grade (E. Merck, F.R. Germany); phenol AR, disodium ethylenediaminetetraacetate AR (Riedel-DE Haën AG, F.R. Germany) ; glycerine U.S.P.XIX (Rubia Industries Ltd., Thailand) ; poncoau 4R (Daiwa, Japan) ; ammonium chloride AR (BDH Chemicals Ltd., Poole, England) ; White Rose of Siam (Charabot & Co., France).

Indicators : Bromocresol purple Lab. grade (May & Baker Ltd., England) ; eriochrome black T indicator (E. Merck, F.R. Germany).

Apparatus

50-ml burette, 250-ml and 500-ml erlenmeyer flask, Beckman Zeromatic SS-3 pH meter with glass electrode (Beckmann Instrument Inc., U.S.A.).

Methods

Preparation of Calamine Lotion

Formula I : Calamine 10 g and zinc oxide 5 g were accurately weighed and 100 ml of lotion was prepared according to U.S.P.XX method (1) using purified water in place of calcium hydroxide solution. White Rose of Siam 0.16 ml and Poncoau 4R 1 mg were used as flavouring and colouring agent respectively.

Formula II : Calamine lotion B.P. 1980 (2) 100 ml was accurately prepared having the same flavour and colour as described in formula I.

Assay Procedures

Method A : Acid-base Titration

Accurately transfered 4 ml of lotion into 250-ml Erlenmeyer flask followed by 25.00 ml of 1 N Sulfuric acid VS, warmed to dissolve, and cooled to room temperature. Ammonium chloride 2.5 g was added, swirled to dissolve, and the solution was titrated with 1 N Sodium hydroxide VS using 5 drops of bromocresol purple TS as indicator until blue-violet end point was reached. Each ml of 1 N Sulfuric acid VS is equivalent to 40.69 mg of ZnO.

Method B : Complexometric Titration (3)

Accurately transfered 1 ml of lotion into 500-ml Erlenmeyer flask and 2 ml of diluted hydrochloric acid was added to dissolve, warmed if necessary, cooled and added 200 ml of water followed by 25 ml of ammonia-ammonium chloride buffer TS. The solution was titrated with 0.05 M disodium ethylenediaminetetraacetate VS using 100 mg of Eriochrome Black T indicator (1 in 500 in potassium chloride) as indicator until blue end point was reached. Blank determination was performed by omitting sample. Each ml of 0.05 M disodium ethylenediaminetetraacetate VS is equivalent to 4.069 mg of ZnO.

Method C : Complexometric Titration

This method was performed as described in method B but 25 ml of triethanolamine was added before ammonia-ammonium chloride buffer TS and the solution was cooled to between 3 to 4° C before titration.

Assay of Commercial Calamine Lotion

A brand of marketed calamine lotion was selected and total zinc oxide content was determined by method A, B and C.

RESULTS AND DISCUSSION

Assay results of calamine lotion formula I determined by method A and method B are shown in Table 1.

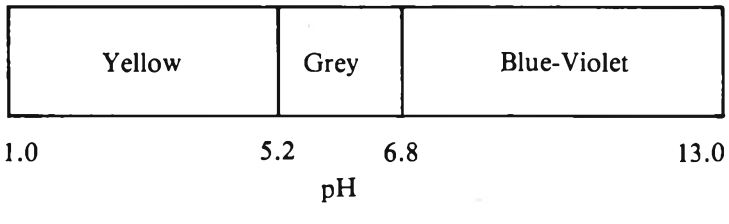
Table 1 Total zinc oxide content in calamine lotion formula I determined by method A and B

Analytical methods	Mean total zinc oxide in 100 ml of lotion			% C.V.	n
	Amount added (g)	Amount found (g)	Percent recovery (%)		
A	11.5775	11.6822	100.90	0.63	11
B	11.5775	12.1661	105.08	0.73	11

pH at end points were within 5.44 ± 0.07 (n = 6).

From Table 1 it appears that mean total zinc oxide content in method B is significantly greater than that of method A with 95 percent of confidence level (t-Test). It is indicated that method A is more accurate than method B.

Method A is acid-base titration, excess sulfuric acid was back titrated with sodium hydroxide. Indicators of choice in the titration of strong acid with strong base must change colour at pH interval of 4.5 to 9.5 (4). In this titration, bromocresol purple was used as indicator and its colour-change is as follows (5):



Colour-change of this indicator from yellow to blue-violet is at pH interval of 5.2 to 6.8 so it conforms with above criterion. The data points out that the pH of end point was 5.44 ± 0.07 (n = 6). Prior to titration the colour of solution was orange-yellow and at the end point the colour changed immediately to blue-violet. Ammonium chloride was added before titration to prevent the reaction of zinc ion in solution with hydroxide ion in the titrant.

Method B is complexometric titration. Bentonite used as suspending agent in formulation caused falsely high value of total zinc oxide content because it is native colloidal, hydrated aluminium silicated and analyais indicates that it is alumino-silicate containing SiO_2, Al_2O_3, MgO and some sodium and

potassium (6) so it can produce many interfering metal ions especially aluminium ion. Namely interference from aluminium ion can be overcome by addition of masking agent such as triethanolamine which appears in method C.

Total zinc oxide contents in lotion Formula I and II determined by method A and C are shown in Table 2.

Table 2 Total zinc oxide contents in calamine lotion formula I and II determined by method A and C.

Analytical methods	Mean total zinc oxide in 100 ml of lotion			% C.V.	n
	Amount added (g)	Amount found (g)	Percent recovery (%)		
Formula I					
Method A	11.3906	11.3032	99.23	0.66	10
Method C	11.3906	11.8282	103.84	0.88	10
Formula II					
Method A	15.7232	15.7555	100.20	0.47	10
Method C	15.7232	16.4482	104.61	0.56	10

From Table 2 it appears that mean total zinc oxide contents in both formulae determined by method C are significantly higher than those determined by method A with confidence level of 95 percent (t-Test). Again method A is more accurate than method C.

A brand of marketed calamine lotion was selected and analysis was performed by method A, B and C. The duplicate results are shown in Table 3.

Table 3 Total zinc oxide contents in calamine lotion (Government Pharmaceutical Organization, Lot. No. G 001029) determined by method A, B and C.

Analytical methods	Mean total zinc oxide in 100 ml of lotion			n
	Amount label (g)*	Amount found (g)	Percent label amount (%)	
Method A	15.5000	14.0258	90.49	2
Method B	15.5000	14.4926	93.50	2
Method C	15.5000	14.4472	93.21	2

* Each 100 ml of lotion contains calamine 15 g and zinc oxide 5 g.
Calculation of calamine is based on 70 percent content of zinc oxide.

From Table 3 it is shown that total zinc oxide contents determined by method B and C are higher than that determined by method A.

CONCLUSION

This experiment indicates that acid-base titration using bromocresol purple as indicator is an appropriated method for total zinc oxide determination in calamine lotion and it is more accurate than complexometric titrations.

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การวิเคราะห์ CALAMINE LOTION

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บทคัดย่อ

การติเตรทแบบกรด-เบสเพื่อหาปริมาณของ zinc oxide รวมในยาน้ำแขวนตะกอน Calamine Lotion ที่มีสีชมพูหรือแดงโดยใช้ bromocresol purple เป็นอินดิเคเตอร์ เป็นวิธีที่ง่าย รวดเร็ว ให้ผลที่ถูกต้องและมีความแม่นยำสูง สามารถสังเกตจุดยุติ (end point) ได้อย่างชัดเจน เหมาะที่จะใช้เป็นวิธีวิเคราะห์ในห้องปฏิบัติการทั่วไป วิธีนี้มีความถูกต้องมากกว่าการติเตรทแบบ complexometric titration (ไทยเภสัชสาร ปีที่ 12 (4) : หน้า 309-314 (2530))

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