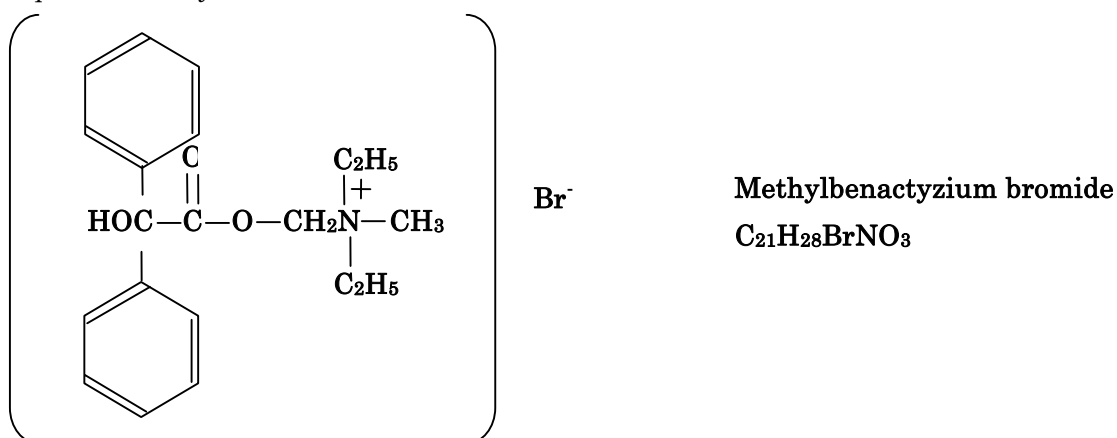


AQUACOUNTER Application Sheet	COM series	DATA No. B1	1st edition
Pharmaceuticals	Quantification of methylbenactyzium bromide by perchloric acid titration		

1. Measurement outline

The method of measuring the purity of antispasmodic methylbenactyzium bromide is specified in Japanese Pharmacopoeia (13th revision) as a quantification method. In this method, the sample is dissolved in acetic anhydride/acetate mixture solvent for potentiometric titration with perchloric acid titrant. 1mol of methylbenactyzium bromide and 1mol of perchloric acid react quantitatively.



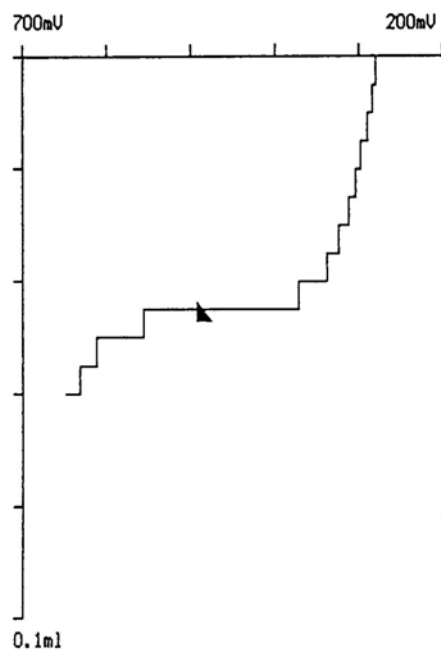
2. Reagents and Electrodes

(1) Reagents	Titrant	0.1mol/L perchloric acid titrant
	Mixture solvent	Acetic anhydride/acetate (4:1) 40mL used for 1 measurement
(2) Electrodes	Indicator electrode	*Glass electrode GE-101B to IE jack
*Standard accessories	Reference electrode	*Reference electrode RE-201 to RE jack
	Note) Inner solution for reference electrode is saturated sodium perchlorate/acetate solution.	

3. Measurement conditions example (for COM-1600S)

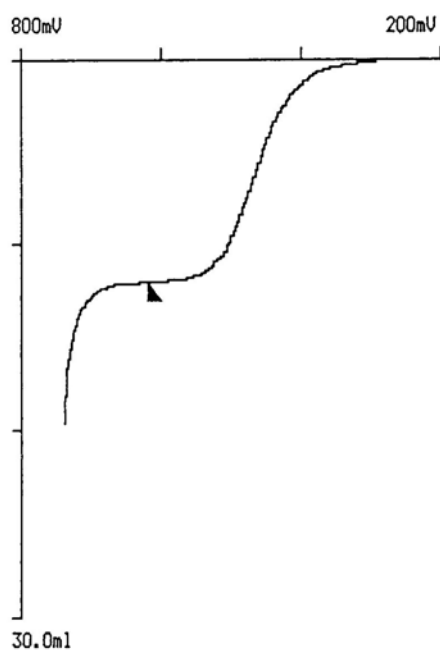
Master File 1							
Condition 1 (for BLANK)				Condition 2 (for measurement)			
Method	AUTO			Method	AUTO		
Amp No.	1	Mode No.	12	Amp No.	1	Mode No.	5
Buret No.	1	Pre Int	0 sec	BURET No.	1	Pre Int	0 sec
Meas Unit	mV	Del K	0	Meas Unit	mV	Del K	5
S-Timer	5 sec	Del Sens	0 mV	S-Timer	5 sec	Del Sens	0 mV
CP mL	0 mL	Int Time	3 sec	CP mL	0 mL	Int Time	3 sec
Direction	N/A	Int Sens	3 mV	Direction	N/A	Int Sens	3 mV
DP mL	0 mL	Brst Speed	2	DP mL	5 mL	Brst Speed	2
End Sens	500	Pulse	4	End Sens	1000	Pulse	40
Over mL	0 mL			Over mL	0 mL		
Max volume	20 mL			Max Volume	20 mL		
Unit	mL			Unit	%		
Blank	0 mL			Blank	BLANK result value		
Factor	0			Factor	Titer of the titrant		
Molarity	0			Molarity	0.1 mol/L		
K	0			K	422.4		
L	0			L	0		
Formula	D			Formula	$(D-B) \times K \times F \times M / (S \times 10)$		

4. Measurement example



Blank measurement results

Sample No.	Titration value (mL)
1	0.042
2	0.046
3	0.044
Avg. (Average value) 0.044 mL	
Std. Dev. (Standard deviation) 0.002 mL	
C.V. (Coefficient of variation) 4.5 %	



Sample measurement results

Sample No.	Sample volume (g)	Titration value (mL)	Concentration (%)
1	0.5003	11.90	100.46
2	0.5000	11.92	100.73
3	0.5006	11.93	100.67
Avg. (Average value)			100.62 %
Std. Dev. (Standard deviation)			0.14 %
C.V. (Coefficient of variation)			0.14 %

5. Outline

(1) In this measurement, the content in the sample is measured accurately and it is necessary that measurement of sample weight, titer of titrant, blank value, etc. be conducted with due caution. It is especially necessary that the titer of the titrant (HClO_4) be measured regularly with potassium hydrogen phthalate. In addition, organic solvent (acetic acid) is used for titrant, and it must be noted that the change in volume in concurrence to temperature change (approximately 0.11% change at 1°C) is larger compared to normal solutions. It is important to try to measure at a stable temperature.

If the concentration of the titrant for titration varies from that of titer evaluation, it is possible to correct it by substituting titer correction formula into the concentration correction formula.

$$F = \frac{F_0}{1 + \alpha(t - t_0)}$$

F	:	Titer at sample titration (after correction)
F_0	:	Titer at titer evaluation
α	:	Volumetric expansion coefficient for titrant (acetic acid = 1.07×10^{-3})
t	:	Temperature for sample titration
t_0	:	Temperature for titer evaluation

(2) Method of preparing reference electrode

The reference electrode to be used in this measurement must be a reference electrode for nonaqueous titration. The following shows the method for preparing the reference electrode to be used for this measurement:

- Release the inner solution for reference electrode RE-201 and wash the inner surface well with acetic acid. Then prepare an acetate solution saturated with sodium perchlorate (special class reagent) and inject it from the refill opening for the electrode. Since the electrode potential may not stabilize immediately, it is recommended that it is used after leaving it standing for 1 entire day.

Key words

Medical product, methylbenactyzium bromide, Japanese Pharmacopoeia, perchloric acid titration, nonaqueous titration, sodium perchlorate saturated/acetic acid solution

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