

Sheet No.

GT200-ME009

Purity Analysis of Anhydrous Caffeine 1/3

Method : Neutralization titration (anhydrous)
 Apparatus : Automatic Titrator GT-200
 Electrode: Reference Electrode, Sleeve Type -Glass Electrode
 Reference electrode inner solution: 3.3 mol/L potassium chloride solution
 Titration mode : INF, Detection: mV
 Related standard : Japanese Pharmacopoeia Anhydrous caffeine, Purity test

*This sheet is provided as information. It is not to guarantee the analysis values. Please use under the ideal conditions considering external factors including the analysis environment and properties of the sample.

Outline

Standards of anhydrous caffeine are stipulated by the Japanese Pharmacopoeia, which describes that 98.5% or greater of anhydrous caffeine is to be contained when quantifying a dried product. Anhydrous caffeine is used for painkiller, energy drinks and other products.

Reagents

[Titrant]

■0.1 mol/L perchloric acid acetic acid solution

[Reagents]

■Acetic anhydride: acetic acid (6:1)

■Reference material for volumetric analysis: Potassium hydrogen phthalate

■Acetic acid

Analytical Procedure

[Blank measurement of acetic acid used for titer evaluation]

- (1) Place 50 ml of acetic acid (special grade) into a 100-ml beaker using a measuring cylinder.
- (2) Titrate using 0.1 mol/L perchloric acid acetic acid solution.

[Titer evaluation of perchloric acid acetic acid solution using potassium hydrogen phthalate]

- (1) Accurately measure 0.3 g of potassium hydrogen phthalate in a 100-ml beaker.
- (2) Add 50 ml of blank-measured acetic acid into the beaker using a measuring cylinder.
- (3) Stir until dissolved, and titrate using 0.1 mol/L perchloric acid acetic acid solution.

[Titer evaluation equation]

Perchloric acid acetic acid solution factor = $1000 / S / ((A1-BL) \times M \times E \times FW)$

S : Sample amount (g)

A1 : Titer of 0.1 mol/L perchloric acid acetic acid solution in titer evaluation (ml)

BL : Blank of acetic acid

M : Molarity of 0.1 mol/L perchloric acid acetic acid solution

E : Equivalent number of 0.1 mol/L perchloric acid acetic acid solution

FW : Formula weight of potassium hydrogen phthalate (204.2)

Sheet No.

GT200-ME009 Purity Analysis of Anhydrous Caffeine

2/3

[Blank measurement of acetic anhydride:acetic acid (6:1) used for sample measurement]

- (1) Place 70 ml of titration solvent into a 100-ml beaker using a measuring cylinder.
- (2) Titrate using 0.1 mol/L perchloric acid acetic acid solution.

[Purity test of anhydrous caffeine]

- (1) Accurately weigh 0.4 g of anhydrous caffeine in a 100-ml beaker to the digit of tenth of a mg.
- (2) Add 70 ml of acetic anhydride:acetic acid (6:1) into the beaker using a measuring cylinder.
- (3) Stir until the anhydrous caffeine is completely dissolved.
- (4) Titrate using 0.1 mol/L perchloric acid acetic acid solution.

[Equation]

Purity (%) = (A1 – BL) × M × E × f × FW / S × R / 10 (using fixed equation)

A1 : Titer of 0.1 mol/L perchloric acid acetic acid solution to the inflection point (ml)

BL : Blank of acetic anhydride:acetic acid (6:1)

M : Molarity of 0.1 mol/L perchloric acid acetic acid solution

E : Equivalent number of 0.1 mol/L perchloric acid acetic acid solution (1)

FW : Formula weight of anhydrous caffeine (194.19)

S : Sample amount (g)

R : Dilution rate (1)

Other

- Wash electrodes with pure water before each measurement, and submerge in pure water for 5 minutes to depolarize.
- Set the buret suction position to 75%.
- Submerge the depolarized electrodes into sample solution after replacing water by pouring alcohol over or wiping water with paper etc.
- Ensure to dry potassium hydrogen phthalate and anhydrous caffeine before use.
- Handle measurement reagents with care after reading through and understanding their labels and safety data sheets.
- Wear personal protective equipment such as protective goggles and gloves when handling the reagents.

Measurement Results

	Sample amount (g)	Titer (ml)	Measurement value (%)
1	0.4040	21.0721	100.3
2	0.4034	21.0161	100.2
3	0.4059	21.1606	100.3

Number of data	(n)	3
Average		100.3
Standard deviation	(SD)	0.0594
Relative standard deviation	(RSD%)	0.0592

Purity of anhydrous caffeine (commercial special grade reagent) was measured. The measured value by GT-200 was 100.3%. Relative standard deviation (RSD%) was 0.06%, exhibiting measurement with relatively high reproducibility.

Sheet No.

GT200-ME009 Purity Analysis of Anhydrous Caffeine

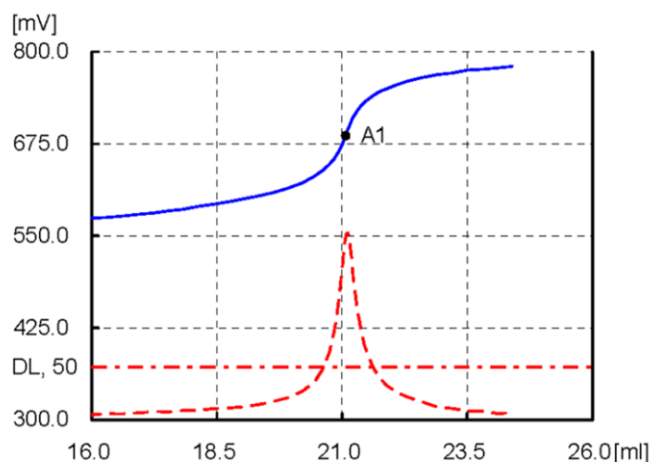
3/3

ID No.: 9 GT No.1

User: GT-200

Measurement date: 2013/12/13 15:16
 Sample name : Anhydrous caffeine

Measurement type : Sample Titr
 Sample size (S) : 0.4040 [g]



C1: 100.326 [%]

A1: 21.0721 [ml] 686 [mV]

Pi : 351 [mV]
 Start : 16 [ml] 574 [mV]
 End : 24.398 [ml] 780 [mV] Time: 7' 9"

Run File No. : 13 Purity of anhydrous caffeine

Titration File No.: 5 Caffeine

Mode : INF End1 End1 Width: 600 [mV] ± 300 [mV]

Detect : mV1

Preset

Mode: V

BRT: 1

BRT No. : 1

Vol: 16 [ml]

Wait: 10 [sec]

Reagent : 9

Pos: Titration

WTint : 30 [sec]

Vup : 300 [μl]

Vlow : 10 [μl]

dE : 2 [mV]

dT : 3 [sec]

DL : 50 [mV/ml]

DetCnt : 20

C1: (A1-BL)*M*E*f*FW/S*R/10

Vmax : 25 [ml]

Vover : 0.3 [ml]

[%]

Reag : 0.1M HClO4/AcOH

E : 1

M : 0.1 [Mol/l]

f : 0.9921

BL : 0.0338 [ml]

FW : 194.19

R : 1

Buret Injection Speed: 500 [ul/sec]