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APPLICATION NOTE

Titratable acidity in orange juice by automatic titration

Water Analysis Instruments, Thermo Fisher Scientific

Key words

TA, Total acidity, citric acid, acidified foods, fruit juice, citrus, grapefruit, lemon, lime, beverage, wine, food, pH, AOAC 942.15, Orion 8172BNWP, Orion 8102BNUMD, Orion Star T910, Orion Star T940.

Introduction

Titratable acidity (TA), as citric acid, in orange juice is determined using the preprogrammed method T1 TitraAcidity. This method is a direct titration to a preset endpoint at pH 8.2 using 0.1M (0.1N) sodium hydroxide titrant. The method may be edited to perform titratable acidity in other samples as well.

Recommended equipment

- Thermo Scientific[™] Orion Star[™] T910 pH Titrator or T940 All-In-One Titrator or equivalent
- Thermo Scientific[™] Orion[™] ROSS[™] SureFlow[™] pH electrode (Cat. No. 8172BNWP) or equivalent
- Orion Automatic Temperature Compensation (ATC) probe
- Analytical balance (for sample measurement by weight) or graduated 10 ml pipet (for sample measurement by volume)



Required reagents and solutions

- Purchased or prepared sodium hydroxide (NaOH) standard titrant solution, 0.1 M (0.1N)
- Reagent grade water (RGW)
- pH buffers: pH 4, 7, and 10

Optional:

• Potassium hydrogen phthalate (KHP) acidimetric standard

Use suitable personal protective equipment (PPE) as recommended by the Safety Data Sheets (SDS) for the chemicals utilized during this procedure.



Titrator setup

Connect the Orion pH electrode, ATC, and the stirrer probe to the titrator. If not previously done, import the T1 TitraAcidity preprogrammed method into the titrator from the Methods screen¹. Rinse and fill the burette with 0.1M (0.1N) sodium hydroxide titrant. See the titrator user manual for details. If bubbles are visible in the tubing, dispense titrant (from the Burette screen) until the bubbles have been expelled. Consider standardizing the titrant before titrating samples. See the following Titrant section.

T1 TitraAcidity Method: Preprogrammed parameters

Electrode	Parameter
Electrode Type	рН
Electrode Name	Edit as desired
Resolution	0.01
Buffer Group	USA

Titrant	Parameter
Titrant Name	NaOH
Titrant ID	
Conc. Input Mode	Standardization
Nominal Concentration	0.1M
Standardize Tech	Equivalence Pt.
Number of Endpoints	1
Results Units	М
Standardize Reaction Ratio	1
Standard Name	KHP
Standard Amount	Variable weight
Standard Molecular Wt	204.2
Standard Purity	100%
Pre-dose Titrant Volume	0 ml
Max. Total Titrant Volume	5 ml
Stand. Process Control	Routine
Pre-stir Duration	5 sec
Stir Speed	Medium

Titration	Parameter
Titration Technique	Preset End Pt.
Number of Endpoints	1
Endpoint Values	8.2
Titration Type	Direct
Blank Required	No
Result Units	%w/w
Reaction Ratio	0.333
Sample Mol. Wt.	192.1
Sample Amount	Variable weight
Pre-dose Titrant Volume	0 ml
Max total titrant volume	10 ml
Titration Process Control	Routine
Pre-stir Duration	5 sec
Stir Speed	Medium
Sample ID	Manual



Electrode preparation

Remove electrode from storage solution. Top up the fill solution to the bottom of the fill hole and leave the fill hole open during testing. Rinse thoroughly with RGW before and between titrations.

Sample preparation

Place a clean 100 or 150 mL beaker on a balance and tare it. Add about 3 grams of orange juice sample to the beaker and record the exact weight to 0.001g or better. Add RGW to the 60 mL mark on the beaker. The sample is ready to titrate.

Sample titration

- From the Home screen, select option to use a saved method, then select the T1 TitraAcidity reprogrammed method.
- 2. At the pre-titration screen, select the Calibrate option and calibrate the electrode with pH 4, 7, and pH 10 buffers.
- After calibration, place the electrode, ATC, stirrer, and dispenser into the sample in the beaker. Ensure that the dispenser tip is inserted below the surface of the sample and start the titration.
- 4. When prompted, enter the exact weight of the sample.

Results

Parameter	Sample	Average (n = 4)	RSD	Analysis Time
Titratable Acidity (as citric acid)	Orange Juice	0.710% (w/w)	0.28%	1.1 minutes ²

Range

This preprogrammed titration method covers a range from 0.5 to 2% acid by weight as citric acid. See below for method modifications to run other concentrations.

Method modifications

- For other concentrations: For less acidic samples, use double the weight of sample. For more acidic samples, use half the weight of sample or change the maximum titration volume to 20 mL by editing the Titration section of the method.
- For other result units: Edit the Titration section of the method and choose the desired unit. If volume-based units are chosen (i.e., % w/v or g acid/100 mL), choose a fixed volume of 3 mL.
- For shorter titrations: For routine titrations with wellestablished endpoint volumes, use a pre-dose to shorten the analysis time. Edit the pre-dose in the Titration section of the method. In general, set the pre-dose at a volume that is 0.5 mL less than the expected endpoint volume.

Titrant

Over time, standard titrant solutions age and can change concentration. For higher accuracy, determine the exact concentration by standardizing the titrant. It is common to standardize on a weekly basis, but other standardization frequencies may be suitable.

- 1. Standardizing the Titrant
 - a. Weigh about 0.05 g KHP into a clean 100 or 150 mL beaker. Record the exact weight to the nearest 0.0001g. Repeat twice more for a total of three beakers of KHP. Add RGW to the 60 mL mark on each beaker and stir for about 2 minutes or so until the KHP is completely dissolved.
 - b. If the KHP purity is not 100%, edit the Titrant section of the method to enter the actual purity
 - c. Select the Titratable Acidity preprogrammed method on the titrator.
 - d. At the pre-titration screen, select the Standardize option and follow the prompts to standardize the titrant.
 - e. The new standardized titrant concentration will automatically be saved and used for subsequent T1 TitraAcidity method titrations.

- 2. Certified Standardized Titrant Solutions
 - a. Some customers may prefer not to standardize their titrant, instead choosing to purchase and use certified standardized titration solutions. In this case, edit the Titrant section of the method and enter the certified concentration and titrant ID (i.e., lot number, if desired).

Titrator and electrode care

Refer to the titrator and electrode user manuals for details on cleaning, storage, and maintenance recommendations to keep the titrator and electrode performing well. Main points for care are summarized as follows.

Daily Care	Weekly or Biweekly Care	As Needed
 If bubbles are visible in the titrator tubing, dispense titrant until bubbles have been expelled Top up the electrode fill solution and leave the fill hole open during measurement Rinse electrode well with RGW between titration cycles Cover the fill hole and store electrode in storage solution overnight 	 Drain and replace the fill solution of the electrode. Change the storage solution in the electrode storage bottle Consider standardizing the titrant on a weekly basis 	 For slow or drifty electrode response, soak 15 minutes in 1% laboratory detergent while stirring. Rinse well with RGW afterwards If still slow or drifty, use Orion pH cleaning solution D per instructions See the user manuals for maintenance details

Notes

¹Refer to the user manual for detailed instructions, if desired.

²With a suitable pre-dose, as described in the Method Modifications section.

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Product	Description	Cat. No.
Titrator kits	Thermo Scientific Orion Star T910 titrator standard kit with 8102BNUWP Thermo Scientific™ Orion™ ROSS Ultra™ pH electrode and ATC probe	
	Orion Star T910 pH titrator SureFlow kit with 8172BNWP ROSS SureFlow pH electrode and ATC probe	START9102
	Orion Star T940 All-In-One titrator standard kit with 8102BNUWP ROSS Ultra pH electrode and ATC probe	START9401
	Orion Star T940 All-In-One titrator SureFlow kit with 8172BNWP ROSS SureFlow pH electrode and ATC probe	START9402
Titrators	Thermo Scientific Orion Star T910 pH Titrator without electrode	START9100
	Thermo Scientific Orion Star T940 All-In-One titrator without electrode	START9400
Electrodes	Thermo Scientific Orion ROSS SureFlow pH Electrode	8172BNWP
	Orion ROSS Ultra pH Electrode	8102BNUWP
	Automatic Temperature Compensation (ATC) probe	927007MD
pH Buffers	Orion pH 4.00 buffer, NIST traceable, 475 ml	910104
	Orion pH 7.00 buffer, NIST traceable, 475 ml	910107
	Orion pH 10.00 buffer, NIST traceable, 475 ml	910110
Reagent Grade Water	Thermo Scientific™ Barnstead™ Smart2Pure™ 12 UV Water Purification System	50129890*
Reagents	0.1M (0.1N) Sodium Hydroxide Titrant	
	Potassium Hydrogen Phthalate, primary or acidimetric standard grade	
Accessories	100 or 150 mL beakers	

^{*}Please contact your local Thermo Scientific representative for support on ordering water quality products. For more information, visit thermofisher.com/waterquality.

References

- Acidity (Titratable) of Fruit Products, Method 942.15. Official Methods of Analysis (OMA). AOAC International, 2275 Research Blvd, Ste 300, Rockville, MD 20850-3250. USA.
- Sadler, G. and Murphy, P. (2010). pH and Titratable Acidity. In: S. Nielsen, ed., Food Analysis, 4th ed. New York: Springer, pp.227-234.

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