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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 $0.1 \mathrm{M}(0.1 \mathrm{~N})$ sodium hydroxide titrant. The method may be edited to perform titratable acidity in other samples as well.

## Recommended equipment

- Thermo Scientific ${ }^{\text {TM }}$ Orion Star ${ }^{\text {TM }}$ T910 pH Titrator or T940 All-In-One Titrator or equivalent
- Thermo Scientific ${ }^{T M}$ Orion ${ }^{T M}$ ROSS $^{T M}$ SureFlow ${ }^{T M} \mathrm{pH}$ 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 $(\mathrm{NaOH})$ standard titrant solution, $0.1 \mathrm{M}(0.1 \mathrm{~N})$
- Reagent grade water (RGW)
- pH buffers: $\mathrm{pH} 4,7$, 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 screen ${ }^{1}$. Rinse and fill the burette with 0.1 M ( 0.1 N ) 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 | pH |
| Electrode Name | Edit as desired |
| Resolution | 0.01 |
| Buffer Group | USA |


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


| 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 0.001 g or better. Add RGW to the 60 mL mark on the beaker. The sample is ready to titrate.

## Sample titration

1. 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 $\mathrm{pH} 4,7$, and pH 10 buffers.
3. 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 <br> $(\mathbf{n}=4)$ | RSD | Analysis <br> Time |
| :--- | :--- | :--- | :--- | :--- |
| Titratable <br> Acidity <br> (as citric <br> acid) | Orange <br> Juice | $0.710 \%$ <br> $(\mathrm{w} / \mathrm{w})$ | $0.28 \%$ | 1.1 <br> 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 0.0001 g . 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

- 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


## Notes

${ }^{1}$ Refer to the user manual for detailed instructions, if desired.
${ }^{2}$ With a suitable pre-dose, as described in the Method Modifications section.

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To purchase Thermo Scientific laboratory products, please contact your local equipment distributor and reference the part numbers listed below:

| Product | Description | Cat. No. |
| :---: | :---: | :---: |
| Titrator kits | Thermo Scientific Orion Star T910 titrator standard kit with 8102BNUWP Thermo Scientific ${ }^{T M}$ Orion ${ }^{T M}$ ROSS Ultra ${ }^{T M} \mathrm{pH}$ electrode and ATC probe | START9101 |
|  | 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 ${ }^{\text {TM }}$ Barnstead $^{\text {TM }}$ Smart2Pure ${ }^{\text {TM }} 12$ UV Water Purification System | 50129890* |
| Reagents | $0.1 \mathrm{M}(0.1 \mathrm{~N})$ 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.


## Find out more at thermofisher.com/waterquality

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