# Titration and Assay Worksheet

Acidimetric Determination of Acetic Acid Content — Acid–Base Titration

**Worksheet Identification**

| | | | |
|---|---|---|---|
| **Document ID** | MAL-QC-TW-0142 | **SOP Reference** | MAL-SOP-014, Rev. 5 |
| **Sample ID** | NC-2247-B-01 | **Batch / Lot** | NC-2247-B |
| **Sample Description** | Vinegar process stream, clarified, Nexa Chemicals fermentation line 2 | | |
| **Analyst** | Priya Anand | **Reviewer** | Marcus Whitfield |
| **Date Performed** | 2026-03-11 | **Date Reviewed** | 2026-03-12 |
| **Balance ID** | BAL-07 (cal. due 2026-09-02) | **Burette ID / Class** | BUR-14, Class A, 50.00 mL |
| **Ambient Temp.** | 21.4 °C | **Relative Humidity** | 46 % |

This worksheet records the standardization of a sodium hydroxide titrant against a primary standard and its subsequent use to assay the acetic acid content of a process-stream sample, in accordance with **MAL-SOP-014**. All volumetric readings are recorded to the nearest 0.01 mL directly from Class A glassware; no readings are back-filled or estimated after the titration is complete.

## 1 Method Summary

### 1.1 Principle

The concentration of a sodium hydroxide (NaOH) titrant is established by direct titration against potassium hydrogen phthalate (KHP), a primary-standard acid, following the general acid–base titrimetry principles described by Harris and Lucy [2015]. The standardized titrant is then used to determine the acetic acid ($CH_3COOH$) content of a diluted vinegar process stream by titration to the phenolphthalein endpoint, consistent with the approach of AOAC Official Method 930.35 [AOAC International, 2019].

$$KHC_8H_4O_4\,(aq) + NaOH\,(aq) \longrightarrow NaKC_8H_4O_4\,(aq) + H_2O\,(l)$$

$$CH_3COOH\,(aq) + NaOH\,(aq) \longrightarrow CH_3COONa\,(aq) + H_2O\,(l)$$

### 1.2 Reagents and Materials

| Reagent | Grade / Supplier | Preparation / Storage |
|---|---|---|
| Sodium hydroxide titrant | ACS grade, Synapse Chemical Supply | 0.1 M nominal, boiled $CO_2$-free water; stored in amber bottle fitted with a soda-lime guard tube |
| Potassium hydrogen phthalate (KHP) | ACS primary standard, 99.95%, Vertex Reagents | Dried 2 h at 110 °C, cooled in desiccator before weighing |
| Phenolphthalein indicator | 1% w/v in 95% ethanol | Amber dropper bottle; 2–3 drops per titration |
| Distilled water | ASTM Type II | In-house purification unit, glass carboy |
| Sample | Vinegar process stream | Nexa Chemicals Batch NC-2247-B; diluted 1:10 with distilled water prior to titration |

> ⚠ **Safety note:** NaOH titrant and KHP dust are corrosive/irritant. Wear eye protection and nitrile gloves throughout. Dispose of neutralized titration waste via the aqueous inorganic waste stream per MAL-SOP-002.

### 1.3 Procedure Outline

1. Dry KHP for 2 h at 110 °C; cool in a desiccator.
2. Weigh three individual KHP portions of approximately 0.51 g into 250 mL Erlenmeyer flasks; dissolve each in 50 mL $CO_2$-free water.
3. Add 2–3 drops of phenolphthalein and titrate each portion with NaOH from a Class A burette to the first persistent pale-pink endpoint (holds $\geq$ 30 s).
4. Dilute 10.00 mL of the vinegar sample to 100.00 mL with distilled water in a volumetric flask.
5. Titrate four 10.00 mL aliquots of the diluted sample with the standardized NaOH, using phenolphthalein as above.
6. Record all initial and final burette readings directly in Section 2 and Section 3 before any calculation is performed.

## 2 Standardization of NaOH Titrant

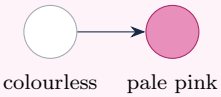
Three independent portions of dried KHP were titrated to the phenolphthalein endpoint. The titrant molarity for each replicate is calculated from $M_{\mathrm{NaOH}} = \dfrac{m_{\mathrm{KHP}}}{MW_{\mathrm{KHP}} \times V_{\mathrm{NaOH}}}$, using $MW_{\mathrm{KHP}} = 204.22\ \mathrm{g\,mol^{-1}}$.

| Rep. | KHP Mass (g) | Initial (mL) | Final (mL) | Vol. Disp. (mL) | $M_{\mathrm{NaOH}}$ (mol/L) |
|---|---|---|---|---|---|
| 1 | 0.5104 | 0.15 | 25.32 | 25.17 | 0.09930 |
| 2 | 0.5088 | 0.20 | 25.10 | 24.90 | 0.10007 |
| 3 | 0.5122 | 0.10 | 25.38 | 25.28 | 0.09922 |

**Standardization Statistics**

| | |
|---|---|
| Mean $M_{\mathrm{NaOH}}$ | 0.09953 M |
| Std. deviation ($s$) | 0.00047 |
| % RSD | 0.47 % |
| $n$ | 3 |
| Acceptance criterion | RSD $\leq$ 1.0 % |
| **Outcome** | **PASS** |

**Endpoint Colour Note**




Endpoint judged at the first *persistent* pale-pink tint, held for $\geq$ 30 s under white bench lighting. All three replicates matched a common Meridian colour-reference card (shade PP-2).

## 3 Sample Assay — Acetic Acid Content

A 10.00 mL aliquot of Batch NC-2247-B was diluted to 100.00 mL with distilled water. Four 10.00 mL aliquots of this diluted solution were titrated in replicate with the standardized NaOH titrant (0.09953 M, Section 2).

| Rep. | Initial (mL) | Final (mL) | Vol. Dispensed (mL) |
|---|---|---|---|
| 1 | 0.12 | 8.44 | 8.32 |
| 2 | 0.18 | 8.55 | 8.37 |
| 3 | 0.10 | 8.51 | 8.41 |
| 4 | 0.15 | 8.47 | 8.32 |

**Replicate Mean & SD**

| | |
|---|---|
| Mean $V_{NaOH}$ | 8.355 mL |
| Std. deviation ($s$) | 0.044 mL |
| % RSD | 0.52 % |
| $n$ | 4 |
| Acceptance criterion | RSD $\leq$ 2.0 % |
| **Outcome** | **PASS** |

**Endpoint Colour Note**

Endpoint: sharp transition from colourless to a persistent **pale pink** (Meridian shade PP-2), stable for $\geq$ 30 s. No fading observed on standing to 2 min, indicating no residual $CO_2$ interference. Aliquots titrated under identical lighting to the standardization series for colour-match consistency.

# 4 Calculations

## 4.1 Titrant Molarity

For each standardization replicate, the NaOH molarity follows from the KHP mass and volume dispensed:

$$M_{NaOH} = \frac{m_{KHP}\,(\text{g})}{MW_{KHP}\,(204.22\,\text{g mol}^{-1}) \times V_{NaOH}\,(\text{L})}$$

Applying this to Replicate 1: $M_{NaOH} = \dfrac{0.5104}{204.22 \times 0.02517} = 0.09930\,\text{M}$, consistent with the three values reported in Section 2 (mean 0.09953 M, RSD 0.47 %), which follows the propagation treatment for replicate titrations described in Skoog et al. [2018].

## 4.2 Acetic Acid Content

The mass of acetic acid ($MW = 60.05\,\text{g mol}^{-1}$) titrated in each 10.00 mL aliquot of the *diluted* sample is:

$$n_{acid} = V_{NaOH} \times M_{NaOH}$$

Using the mean dispensed volume from Section 3:

$$n_{acid} = 8.355\,\text{mL} \times 0.09953\,\text{M} = 0.8316\,\text{mmol}$$

Converting to concentration in the diluted solution and correcting for the 1:10 dilution factor ($DF = 10$) recovers the concentration in the original process stream:

$$C_{acid,\,original} = \frac{n_{acid}}{V_{aliquot}} \times DF = \frac{0.8316\,\text{mmol}}{10.00\,\text{mL}} \times 10 = 0.8316\,\text{M}$$

Expressed as % w/v acetic acid:

$$\%\,\text{w/v} = \frac{C_{acid,\,original} \times MW_{acid}}{10} = \frac{0.8316 \times 60.05}{10} = 4.99\,\%\ \text{w/v}$$

**Result**

Acetic acid content of Batch NC-2247-B: **4.99 % w/v** (label claim 5.00 % ± 5 % relative)

# 5 Outlier Check and Result Summary

## 5.1 Outlier Screening

The sample-assay replicate volumes (Section 3) were screened with the Dixon $Q$-test at 90% confidence ($Q_{crit} = 0.831$ for $n = 4$). The most extreme value, Replicate 3 (8.41 mL), gives:

$$Q = \frac{|8.41 - 8.37|}{8.41 - 8.32} = \frac{0.04}{0.09} = 0.444$$

Since $Q < Q_{crit}$, no replicate is rejected as a statistical outlier; all four values are retained in the reported mean.

### 5.2 Result Summary

| Parameter | Result | Acceptance Criterion | Outcome |
|---|---|---|---|
| NaOH standardized molarity | 0.09953 M | RSD $\leq$ 1.0 % (got 0.47 %) | PASS |
| Sample replicate precision | 0.52 % RSD | RSD $\leq$ 2.0 % | PASS |
| Outlier check ($Q$-test) | $Q = 0.444$ | $Q < 0.831$ | PASS |
| Acetic acid content | 4.99 % w/v | 4.75 % to 5.25 % w/v | PASS |

**Analyst Comments**

Titrations proceeded without incident; burette tip checked for air bubbles before each replicate. Endpoint colour matched the Meridian PP-2 reference card across all seven titrations (standardization and assay combined). Result falls within label-claim tolerance; no re-titration required.

### 5.3 Sign-off

| Role | Name / Signature | Date |
|---|---|---|
| Analyst | Priya Anand _____ | 2026-03-11 |
| Reviewer | Marcus Whitfield _____ | 2026-03-12 |
| QA Approval | _____ | _____ |

## References

AOAC International. Official method 930.35: Acidity (titratable) of vinegar. Aoac official methods of analysis, AOAC International, 2019.

Daniel C. Harris and Charles A. Lucy. *Quantitative Chemical Analysis*. W. H. Freeman, New York, NY, 9th edition, 2015.

Douglas A. Skoog, Donald M. West, F. James Holler, and Stanley R. Crouch. *Fundamentals of Analytical Chemistry*. Cengage Learning, Boston, MA, 9th edition, 2018.