

### Operational quality performance Check: Nitrogen determination in reference substance

*Kjel Line and MultiDist:*

*Nitrogen determination in ammonium dihydrogen phosphate salt by direct distillation*

#### 1. Introduction

Performance check with a reference substance is a key element in qualifying a distillation unit in an operational qualification (OQ) procedure. To verify the Kjel Line and MultiDist instruments the nitrogen content of dry ammonium dihydrogen phosphate monobasic (ADP) is determined according to Kjeldahl method. Direct distillation with the steam distillation unit MultiDist was followed by boric acid titration on the Metrohm Eco Titrator. Alternatively, manual titration with/without a titrator could also be performed to quantify the distilled Nitrogen content.

#### 2. Experiment

##### Equipment:

MultiDist coupled with Metrohm EcoTitrator (11K36532210)

##### Procedure:

Weigh 0.2g reference substance ammonium dihydrogen phosphate (assay=99.8%). Distillation and Titration parameters are as presented in Table 2. (For detailed procedure please download AN751/2021)

Table 1. Distillation parameters on Distillation unit

Parameter	Direct Distillation
H <sub>2</sub> O Volume	35mL
NaOH Volume	40mL
Distillation Time	180s
Titration Type	Boric Acid Titration
H <sub>3</sub> BO <sub>3</sub> Volume	60 mL (4%)
Sensor type	Potentiometric (pH)
Endpoint pH	4.65
Titrant	H <sub>2</sub> SO <sub>4</sub> 0.1mol/L



#### 3. Results

All measured recovery rates for ADP were within specifications of 99.5-102%.

Table 3: Determined Nitrogen contents (rsd in brackets, n=5).

Product	Recoveries (RSD)	Instrument
Ammonium dihydrogen phosphate 1	100.21%	MultiDist
Ammonium dihydrogen phosphate 2	100.43%	MultiDist
Ammonium dihydrogen phosphate 3	100.50%	MultiDist
Ammonium dihydrogen phosphate 4	100.52%	MultiDist
Ammonium dihydrogen phosphate 5	100.39%	MultiDist
Average	100.41% (0.1%)	

#### 4. Conclusion

The presented procedure provides an easy and effective way to check the performance of the distillation unit. The obtained recovery rates on steam distillation followed by titration as described in the Application Note should fall within 99.5-102% for ammonium dihydrogen phosphate.

#### 5. References

AN751/2021, Operation Manual Kjel-/DistLine