Dow AgroSciences

Analytical Method

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Version: 1

DETERMINATION OF WATER CONTENT BY KARL FISCHER

1. SCOPE

The procedure described in this method is suitable for the determination of water content of pesticide formulations. This method is intended to meet or exceed standards set by performance test method CIPAC MT 30.1.

2. PRODUCT SAFETY PRECAUTIONS

Each analyst should be acquainted with potential hazards of the reagents, products and solvents before commencing laboratory work. SOURCES OF INFORMATION INCLUDE: MATERIAL SAFETY DATA SHEETS, LITERATURE AND OTHER INTERNALLY GENERATED DATA. Safety information on non-Dow AgroSciences products should be requested from the supplier. Disposal of reagents, reactants and solvents must be in compliance with the appropriate government regulations.

3. PRINCIPLE

Volumetric Procedure
The sample is dispersed in methanol, and titrated with standard Karl Fischer reagent of known water equivalent.

Coulometric Procedure
Samples are injected directly into the Karl Fischer vessel, and any water is then automatically titrated by electrically produced iodine in the coulometric cell.

4. APPARATUS

Standard Karl Fischer equipment from various suppliers

5. REAGENTS

Volumetric Procedure
Methanol (CH_3OH) containing not more than 0.03% water m/m; RE 47
Karl Fischer reagent (Note 8.1)

Coulometric Procedure
Analyte and catholyte as recommended by the instrument's manufacturer.

6. PROCEDURE

Volumetric Procedure
Add the methanol (about 20 mL) to the titration vessel and titrate to the nullpoint with Karl Fischer reagent. Transfer quickly a suitable amount of sample (about 2 g), accurately weighed (*w* g), into the titration vessel. Stir for 1 min and titrate again with the standardized Karl Fischer reagent (*t* mL) of known water equivalent.

Coulometric Procedure
Press the start button of the titrator, and then add a known amount of the sample to the Karl Fischer (KF) titration vessel. The sample size will depend on the amount of water in the sample. Typically 0.05 to 0.5 grams is sufficient. Liquid samples are usually measured into the titration vessel by means of a syringe. If this is not possible, they can be added to a quantity of auxiliary solution (such as methanol) whose water content has already been determined. Solid substances can be introduced through the top of the titration vessel and should be as finely powered as possible. Alternatively, the water from solid samples can be extracted or dissolved into an appropriate solvent with known, or concurrently determined, moisture content. After entering the weight of the sample added to the KF titration vessel, the water content will be calculated by the instrument and printed and/or displayed.

7. CALCULATIONS

Volumetric Procedure
Calculate the water content using the following formula:

$$\text{Water Content} = \frac{t \text{ x } e}{10\ w} \text{ \% m/m}$$

where:
e = water equivalent in mg/ml of Karl Fischer reagent

8 NOTES

8.1 Karl Fischer reagent deteriorates rapidly and should be standardized immediately before use, or daily, as required. When freshly prepared, 1 mL is equivalent to about 5 mg of water. Reagents of suitable concentration are commercially available and despite their high costs, are cheaper than making the solution oneself, as this requires a lot of experience for obtaining reliable results.

8.2 Additional test requirements may be detailed by a production specification.

9 REFERENCES

9.1. CIPAC Handbook, Volume F, Physico-chemical Methods for Technical and Formulated Pesticides, MT 30.1

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