FAO SPECIFICATIONS AND EVALUATIONS

FOR AGRICULTURAL PESTICIDES

ENDOSULFAN

(1,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3ylenebismethylene) sulfite



FOOD AND AGRICULTURE ORGANIZATION of THE UNITED NATIONS

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DISCLAIMER¹

FAO specifications are developed with the basic objective of promoting, as far as practicable, the manufacture, distribution and use of pesticides that meet basic quality requirements.

Compliance with the specifications does not constitute an endorsement or warranty of the fitness of a particular pesticide for a particular purpose, including its suitability for the control of any given pest, or its suitability for use in a particular area. Owing to the complexity of the problems involved, the suitability of pesticides for a particular purpose and the content of the labelling instructions must be decided at the national or provincial level.

Furthermore, pesticides which are manufactured to comply with these specifications are not exempted from any safety regulation or other legal or administrative provision applicable to their manufacture, sale, transportation, storage, handling, preparation and/or use.

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¹ This disclaimer applies to all specifications published by FAO.

PART ONE

SPECIFICATIONS

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ENDOSULFAN

INFORMATION

ISO common name

endosulfan (BSO, E-ISO, (m) F-ISO, ANSI, ESA)

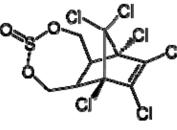
Synonyms

thiodan, benzoepin

Chemical names

- *IUPAC* (1,4,5,6,7,7-hexachloro-8,9,10-trinorborn-5-en-2,3-ylenebismethylene) sulfite
- *CA* 6,7,8,9,10,10-hexachloro-1,5,5a,6,9,9a-hexahydro-6,9-methano-2,4,3benzodioxathiepin 3-oxide

Structural formula



Molecular formula

 $C_9H_6CI_6O_3S$

Relative molecular mass

406.96

CAS Registry number

endosulfan:	115-29-7
alpha-endosulfan:	959-98-8
beta-endosulfan:	33213-65-9

CIPAC number

89

Identity tests

GC retention time, IR spectrum, EI mass spectrum (from GC-MS)

ENDOSULFAN TECHNICAL MATERIAL

FAO specification 89/TC/S (updated on March 2011^{*})

1 **Description**

The material shall consist of endosulfan (mixture of the alpha- and betaisomers of endosulfan) together with related manufacturing impurities, and shall be in the form of cream or brown-coloured granules, flakes or powder with the tendency to agglomeration, free from visible extraneous matter and added modifying agents.

2 Active ingredient

2.1 Identity tests (89/TC/M2/2, CIPAC Handbook 1C, p. 2110, 1985)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 Endosulfan content (89/TC/M2/3, CIPAC Handbook 1C, p. 2110, 1985)

The endosulfan content shall be declared (not less than 940 g/kg) and, when determined, the average measured content shall not be lower than the declared minimum content.

2.3 Endosulfan isomers ratio (89/TC/M2/3, CIPAC Handbook 1C, p. 2110, 1985) (Note 1)

The (*alpha:beta*) endosulfan isomer ratio shall be declared and, when determined, the average measured ratio shall be in the range 60:40 to 75:25.

3 Relevant impurities

3.1 Water content (MT 30.5, CIPAC Handbook J, p. 120, 2000)

Maximum: 10 g/kg.

3.2 Material insoluble in acetone (MT 27, CIPAC Handbook F, p. 88, 1995) Maximum: 10 g/kg.

⁶ In 2007 endosulfan was recommended to be included in the Rotterdam Convention on Prior Informed Consent and in the list of chemicals banned under the Stockholm Convention on Persistent Organic Pollutants. This specification is only an update version of the specification developed under the old procedure. No new data were submitted for this compound, nevertheless, as this compound is still used in some countries, the 2010 JMPS decided to maintain the specification instead of withdrawing it.

Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at:

http://www.fao.org/agriculture/crops/core-themes/theme/pests/pm/jmps/ps/en/.

- 4.1 Acidity (MT 31.2.1, CIPAC Handbook F, p. 98, 1995)Maximum acidity: 1 g/kg calculated as H₂SO₄.
- Note 1 The The CIPAC method 89/TC/M2/3 allows the chromatographic separation of the alpha- and beta- endosulfan isomers and therefore the (*alpha:beta*) endosulfan isomer ratio should be calculated using this method.

ENDOSULFAN DUSTABLE POWDER

FAO specification 89/DP/S (updated on March 2011^{*})

1 **Description**

The material shall consist of a homogeneous mixture of technical endosulfan, complying with the requirements of FAO specification 89/TC/S (March 2011), together with carriers and any other necessary formulants. It shall be in the form of a fine, free-flowing powder, free from visible extraneous matter and hard lumps.

2 Active ingredient

2.1 Identity tests (89/DP/M2/2, CIPAC Handbook 1C, p. 2113, 1985)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 Endosulfan content

(89/DP/M2/3, CIPAC Handbook 1C, p. 2113, 1985)

The endosulfan content shall be declared (g/kg) and, when determined, the average content measured shall not differ from that declared by more than $\pm 10\%$.

3 Relevant impurities

3.1 Water content (MT 30.5, CIPAC Handbook J, p. 120, 2000)

Maximum: 20 g/kg.

4 **Physical properties**

4.1 Acidity or alkalinity (MT 31.2.2, CIPAC Handbook F, p. 98, 1995)

Maximum acidity: 1 g/kg calculated as H_2SO_4 .

Maximum alkalinity: 2 g/kg calculated as NaOH.

In 2007 endosulfan was recommended to be included in the Rotterdam Convention on Prior Informed Consent and in the list of chemicals banned under the Stockholm Convention on Persistent Organic Pollutants. This specification is only an update version of the specification developed under the old procedure. No new data were submitted for this compound, nevertheless, as this compound is still used in some countries, the 2010 JMPS decided to maintain the specification instead of withdrawing it.

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http://www.fao.org/agriculture/crops/core-themes/theme/pests/pm/jmps/ps/en/.

- 4.2 Dry sieve test (MT 59.1, CIPAC Handbook F, p. 177, 1995)
- 4.2.1 For crop protection products used on plants

Maximum: 5% retained on a 75 μ m test sieve. Not more than (0.005 x X)% of the mass of the sample used for the determination shall be present as endosulfan in the residue on the sieve, where X is the endosulfan content (g/kg) found under 2.2 (Note 1).

4.2.2 For crop protection products used by ground application, stored products and similar

Maximum: 2% retained on a 150 µm test sieve.

Not more than $(0.002 \times X)\%$ of the mass of the sample used for the determination shall be present as endosulfan in the residue on the sieve, where X is the endosulfan content (g/kg) found under 2.2 (Note 1).

5 Storage stability

5.1 **Stability at elevated temperature**

(MT 46.3, CIPAC Handbook J, p. 128, 2000)

After storage at $54 \pm 2^{\circ}$ C for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined average content found before storage (Note 2) and the formulation shall continue to comply with the clauses for:

- acidity or alkalinity (4.1);
- dry sieve test (4.2).
- Note 1 If the formulation has a found content of 40 g/kg of endosulfan and 20 g of sample is used in the test, then the amount of endosulfan in the residue on the sieve should not exceed 0.040 g for clause 4.2.1, i.e.

<u>Note 2</u> Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

ENDOSULFAN WETTABLE POWDER

FAO specification 89/WP/S (updated on March 2011^{*})

1 **Description**

The material shall consist of a homogeneous mixture of technical endosulfan, complying with the requirements of FAO specification 89/TC/S (March 2011), together with filler(s) and any other necessary formulants. It shall be in the form of a fine powder, free from visible extraneous matter and hard lumps.

2 Active ingredient

2.1 Identity tests (89/WP/M2/2, CIPAC Handbook 1C, p. 2114, 1985)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 Endosulfan content

(89/WP/M2/3, CIPAC Handbook 1C, p. 2114, 1985)

The endosulfan content shall be declared (g/kg) and, when determined, the average content measured shall not differ from that declared by more than the following tolerances:

Declared content, g/kg	Tolerance
up to 500	± 5% of the declared content
above 500	± 25 g/kg
Note: the upper limit is included in each range	

3 Relevant impurities

3.1 Water content (MT 30.5, CIPAC Handbook J, p. 120, 2000)

Maximum: 20 g/kg.

In 2007 endosulfan was recommended to be included in the Rotterdam Convention on Prior Informed Consent and in the list of chemicals banned under the Stockholm Convention on Persistent Organic Pollutants. This specification is only an update version of the specification developed under the old procedure. No new data were submitted for this compound, nevertheless, as this compound is still used in some countries, the 2010 JMPS decided to maintain the specification instead of withdrawing it.

Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at:

http://www.fao.org/agriculture/crops/core-themes/theme/pests/pm/jmps/ps/en/.

- 4.1 Acidity or alkalinity (MT 31.2.2, CIPAC Handbook F, p. 98, 1995) Maximum acidity: 2 g/kg calculated as H₂SO₄.
 Maximum alkalinity: 2 g/kg calculated as NaOH.
- 4.2 Wet sieve test (MT 185, CIPAC Handbook K, p. 149, 2003)Maximum: 2% retained on a 75 µm test sieve.
- 4.3 **Suspensibility** (MT 184, CIPAC Handbook K, p. 142, 2003) (Notes 1 & 2)

A minimum of 50% of the endosulfan content found under 2.2 shall be in suspension after 30 min in CIPAC Standard Water D at $30 \pm 2^{\circ}C$ (Note 3).

4.4 Persistent foam (MT 47.2, CIPAC Handbook F, p. 152, 1995) (Note 4)

Maximum: 25 mL after 1 min.

4.5 Wettability (MT 53.3.1, CIPAC Handbook F, p. 164, 1995)

The formulation shall be completely wetted in 2 min without swirling.

5 Storage stability

5.1 **Stability at elevated temperature**

(MT 46.3, CIPAC Handbook J, p. 128, 2000)

After storage at $54 \pm 2^{\circ}$ C for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined average content found before storage (Note 5) and the formulation shall continue to comply with the clauses for:

- acidity or alkalinity (4.1);
- wet sieve test (4.2);
- <u>Note 1</u> The formulation should be tested at highest and lowest rates of use recommended by the supplier, provided this does not exceed the conditions given in the method.
- Note 2 This test will normally only be carried out after the heat stability test, 5.1.
- <u>Note 3</u> Chemical assay is the only fully reliable method to measure the mass of active ingredient still in suspension. However, simpler methods such as gravimetric and solvent extraction determination may be used on a routine basis provided that these methods have been shown to give equal results to those of chemical assay. In case of dispute, chemical assay shall be the "referee method".
- <u>Note 4</u> The mass of sample to be used in the test should be at the highest rate of use recommended by the supplier. The test is to be conducted in CIPAC standard water D.
- <u>Note 5</u> Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

ENDOSULFAN OIL MISCIBLE LIQUID

FAO specification 89/OL/S (updated on March 2011^{*})

1 **Description**

The material shall consist of technical endosulfan, complying with the requirements of FAO specification 89/TC/S (March 2011), together with any other necessary formulants. It shall be free from visible suspended matter and sediment.

2 Active ingredient

2.1 Identity tests (89/EC/M2/2, CIPAC Handbook 1C, p. 2114, 1985) (Note 1)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 Endosulfan content (89/EC/M2/3, CIPAC Handbook 1C, p. 2114, 1985) (Note 1)

The endosulfan content shall be declared (g/kg or g/l at $20 \pm 2^{\circ}$ C, Note 2) and, when determined, the average content measured shall not differ from that declared by more than the following tolerances:

Declared content, g/kg or g/l at 20 ± 2°C	Tolerance
up to 25	± 15% of the declared content
above 25 up to 100	± 10% of the declared content
Note: the upper limit is included in each range	

3 Relevant impurities

3.1 Water content (MT 30.5, CIPAC Handbook J, p. 120, 2000)

Maximum: 5 g/kg.

In 2007 endosulfan was recommended to be included in the Rotterdam Convention on Prior Informed Consent and in the list of chemicals banned under the Stockholm Convention on Persistent Organic Pollutants. This specification is only an update version of the specification developed under the old procedure. No new data were submitted for this compound, nevertheless, as this compound is still used in some countries, the 2010 JMPS decided to maintain the specification instead of withdrawing it.

Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at:

http://www.fao.org/agriculture/crops/core-themes/theme/pests/pm/jmps/ps/en/.

4.1 Acidity or alkalinity (MT 31.2.3, CIPAC Handbook F, p. 99, 1995)

Maximum acidity: 0.5 g/kg calculated as H_2SO_4 .

Maximum alkalinity: 0.5 g/kg calculated as NaOH.

4.2 Miscibility with hydrocarbon oil (MT 23, CIPAC Handbook F, p. 85, 1995) If required, the formulation shall be miscible with the appropriate hydrocarbon oil (Note 3).

5 Storage stability

5.1 **Stability at 0°C** (MT 39.3, CIPAC Handbook J, p. 126, 2000)

After storage at 0 \pm 2°C for 7 days, the volume of solid and/or liquid which separates shall not be more than 0.3 mL.

5.2 **Stability at elevated temperature**

(MT 46.3, CIPAC Handbook J, p. 128, 2000)

After storage at $54 \pm 2^{\circ}$ C for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined average content found before storage (Note 4) and the formulation shall continue to comply with the clauses for:

- acidity or alkalinity (4.1);
- Note 1 The CIPAC methods 89/EC/M2/2 and 89/EC/M2/3 for identity tests and endosulfan content for EC formulations are applicable to OL formulations as well.
- <u>Note 2</u> If the buyer requires both g/kg and g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.
- <u>Note 3</u> The concentration should not be higher than the highest concentration recommended in the instructions for use.
- <u>Note 4</u> Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

ENDOSULFAN EMULSIFIABLE CONCENTRATE

FAO specification 89/EC/S (updated on March 2011^{*})

1 **Description**

The material shall consist of technical endosulfan, complying with the requirements of FAO specification 89/TC/S (March 2011), dissolved in suitable solvents, together with any other necessary formulants. It shall be in the form of a stable homogeneous liquid, free from visible suspended matter and sediment, to be applied as an emulsion after dilution in water.

2 Active ingredient

2.1 Identity tests (89/EC/M2/2, CIPAC Handbook 1C, p. 2114, 1985)

The active ingredient shall comply with an identity test and, where the identity remains in doubt, shall comply with at least one additional test.

2.2 Endosulfan content (89/EC/M2/3, CIPAC Handbook 1C, p. 2114, 1985)

The endosulfan content shall be declared (g/kg or g/l at $20 \pm 2^{\circ}$ C, Note 1) and, when determined, the average content measured shall not differ from that declared by more than the following tolerances:

Declared content, g/kg or g/l at 20 ± 2°C	Tolerance
above 250 up to 500	± 5% of the declared content
above 500	± 25 g/kg or g/l
Note: the upper limit is included in each range	

3 Relevant impurities

3.1 Water content (MT 30.5, CIPAC Handbook J, p. 120, 2000)

Maximum: 0.5 g/kg.

In 2007 endosulfan was recommended to be included in the Rotterdam Convention on Prior Informed Consent and in the list of chemicals banned under the Stockholm Convention on Persistent Organic Pollutants. This specification is only an update version of the specification developed under the old procedure. No new data were submitted for this compound, nevertheless, as this compound is still used in some countries, the 2010 JMPS decided to maintain the specification instead of withdrawing it.

Specifications may be revised and/or additional evaluations may be undertaken. Ensure the use of current versions by checking at:

http://www.fao.org/agriculture/crops/core-themes/theme/pests/pm/jmps/ps/en/.

4.1 Acidity or alkalinity (MT 31.2.3, CIPAC Handbook F, p. 99, 1995)

Maximum acidity: 0.5 g/kg calculated as H_2SO_4 .

Maximum alkalinity: 0.5 g/kg calculated as NaOH.

4.2 **Emulsion stability and re-emulsification** (MT 36.1.1, CIPAC Handbook F, p. 108, 1995) (Note 2)

The formulation, when diluted at $30^{\circ} \pm 2^{\circ}$ C with CIPAC Standard Waters A and D, shall comply with the following:

Time after dilution	Limits of stability
0 h	Initial emulsification complete
0.5 h	"Cream", maximum: 2 mL
2.0 h	"Cream", maximum: 4 mL "Free oil", none
24 h	Re-emulsification complete
24.5 h	"Cream", maximum: 4 mL "Free oil", maximum: 0.5 mL
Note: tests after 24 h are required only where the results at 2 h are in doubt.	

4.3 **Persistent foam** (MT 47.2, CIPAC Handbook F, p. 152, 1995) (Note 3) Maximum: 60 mL after 1 min in CIPAC Standard Water A.

5 Storage stability

5.1 **Stability at 0°C** (MT 39.3, CIPAC Handbook J, p. 126, 2000)

After storage at 0 \pm 2°C for 7 days, the volume of solid and/or liquid which separates shall not be more than 0.3 mL.

5.2 **Stability at elevated temperature**

(MT 46.3, CIPAC Handbook J, p. 128, 2000)

After storage at $54 \pm 2^{\circ}$ C for 14 days, the determined average active ingredient content must not be lower than 95% relative to the determined average content found before storage (Note 4) and the formulation shall continue to comply with the clauses for:

- acidity or alkalinity (4.1);
- emulsion stability and re-emulsification (4.2).

Note 2 The formulation will be tested at 5% dilution.

Note 1 If the buyer requires both g/kg and g/l at 20°C, then in case of dispute the analytical results shall be calculated as g/kg.

- <u>Note 3</u> The mass of sample to be used in the test should correspond to the highest rate of use recommended by the supplier.
- Note 4 Samples of the formulation taken before and after the storage stability test should be analyzed concurrently after the test in order to reduce the analytical error.

PART TWO

EVALUATION REPORTS

ENDOSULFAN

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2010 FAO/WHO evaluation report based on review of specifications by JMPS

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ENDOSULFAN

FAO/WHO EVALUATION REPORT 89/2010

Recommendations

The Meeting recommended the following.

- (i) The existing WHO specifications for endosulfan TC (WHO/SIT/27.R1, December 1999) and EC (WHO/SIF/49.R1, December 1999) should be withdrawn.
- (ii) The updated WHO specifications for endosulfan TC and EC, as proposed by JMPS, should be adopted by WHO and published under the category of specifications developed under the old procedure.
- (iii) The existing FAO specifications for endosulfan TC (89/TC/S, 1989), DP (89/DP/S, 1989), WP (89/WP/S, 1989), OL (89/OL/S, 1989) and EC (89/EC/S, 1989) should be withdrawn.
- (iv) The updated FAO specifications for endosulfan TC, DP, WP, OL and EC, as proposed by JMPS, should be adopted by FAO and published under the category of specifications developed under the old procedure.

Appraisal

The Meeting considered the review of the existing WHO specifications for endosulfan TC (WHO/SIT/27.R1, December 1999) and EC (WHO/SIF/49.R1, December 1999), and of the existing FAO specifications for endosulfan TC (89/TC/S, 1989), DP (89/DP/S, 1989), WP (89/WP/S, 1989), OL (89/OL/S, 1989) and EC (89/EC/S, 1989).

In 2007 endosulfan was recommended to be included in the Rotterdam Convention on Prior Informed Consent and in the list of chemicals banned under the Stockholm Convention on Persistent Organic Pollutants (POPs). The FAO/WHO Manual on pesticides specifications states that compounds considered as POP chemicals should be re-evaluated. Since the product is still used in agriculture and public health in some countries and no new data were submitted for development of specifications under the requirements of the FAO/WHO new procedure, the Meeting agreed to update, but to maintain, the specifications under the old procedure.

The Meeting considered that the purpose of the reviews is to update the methods only and that clauses in the old specifications should remain. The Meeting agreed to add a footnote against the date of the specification to indicate that the date applies to the date of publication of the specification and that it is only an update version of a specification developed under the old procedure.

The updated specifications include the following changes by comparison with the old specifications.

General considerations

- The updated specifications for endosulfan TC, DP, WP, OL and EC were written according to the format requirements (specification guidelines) of the FAO/WHO Manual (November 2010 second revision of the First Edition).
- Information on endosulfan was added in the updated specifications.
- The methods for endosulfan content extensively described in the old specifications were referenced in the updated specification to the existing CIPAC methods published in Handbook 1C.
- While maintaining the clauses and their respective limits, the methods for physicochemical properties referenced in the old specifications were updated according to the current CIPAC referee methods when applicable.
- The information on packing and marking of packages of the old specifications were withdrawn in the updated specifications because this information is no longer required under the new procedure.
- For acidity / alkalinity, the Meeting agreed to maintain the old CIPAC method MT 31 instead of the new CIPAC method MT 191, because the limit specified may not be achieved with the new method.

Endosulfan technical material (TC)

- The Meeting agreed to maintain in the updated WHO and FAO specifications the minimum endosulfan content as declared in the old specifications (not less than 920 g/kg for WHO specification and not less than 940 g/kg for FAO specification).
- The endosulfan isomers ratio was specified in the revised WHO/FAO specifications instead of the content of each isomer.
- The clause for melting / freezing point of the old WHO/FAO specifications was withdrawn in the updated WHO/FAO specifications because of the existence of an identity test.
- The clauses for impurities endosulfan-ether, endosulfan-alcohol and endosulfan sulphate of the old FAO specification were withdrawn in the updated WHO/FAO specification because no peer validated methods are available. These impurities were not specified in the old WHO specification.
- The clause for loss on drying of the old FAO specification was withdrawn in the updated WHO/FAO specifications because of the existence of a clause for water content.

Endosulfan dustable powder (DP)

- The clause for flowability of the old FAO specification was withdrawn in the updated FAO specification because this test is not planned in the specification guideline for DP of the FAO/WHO Manual (November 2010 - second revision of the First Edition).

Endosulfan wettable powder (WP)

- For the suspensibility, the CIPAC Standard Water C in the old FAO specification was replaced in the updated FAO specification by the CIPAC Standard Water D.

Endosulfan oil miscible liquid (OL)

- The ranges and limits for endosulfan content of the old FAO specification were adapted in the updated FAO specification according to the recommendations of the FAO/WHO Manual (November 2010 second revision of the First Edition).
- For identity tests and endosulfan content, the Meeting agreed to add a footnote explaining that the CIPAC methods 89/EC/M2/2 and 89/EC/M2/3 for EC formulations are applicable to OL formulations as well.

Endosulfan emulsifiable concentrate (EC)

- For the emulsion stability and persistent foam tests, the WHO standard soft and hard waters mentioned in the old specification were replaced in the updated WHO/FAO specifications by the CIPAC Standard Waters A and D respectively. For the emulsion stability test, the CIPAC Standard Water C mentioned in the old FAO specification was replaced in the updated WHO/FAO specification by the CIPAC Standard Water D.
- For the emulsion stability test, the method WHO/M/29 (with examination after only 2 h) of the old WHO specification was replaced in the updated WHO/FAO specification by the CIPAC method MT 36.1.1 (with examination at 0 h, 0.5 h, 2.0 h, 24 h and 24.5 h). The clauses of the old FAO specification were adopted in the updated WHO/FAO specifications.
- The test of flash point of the old WHO/FAO specification was withdrawn in the updated WHO/FAO specification, as recommended by the specification guideline for EC of the FAO/WHO Manual (November 2010 second revision of the First Edition).