

PHOXIM TECHNICAL

Specification WHO/SIT/29
Approved 25 September 1989

1. Specification

1.1 Material

The material shall consist of phoxim together with related manufacturing compounds and shall be in the form of a reddish-brown liquid free from extraneous impurities or added modifying agents other than the stabilizer.

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment (see method WHO/M/1), shall comply with the requirements of section 1.1 and with the following requirements:

1.2.1 *Phoxim content (g/kg basis)*

The phoxim content shall be declared (not less than 840 g/kg) and, when determined by the method described in section 2.1, the content obtained shall not differ from that declared by more than ± 20 g.

1.2.2 *Acidity*

The acidity of the material, determined by the method described in WHO/M/3, shall not be higher than 1 g/kg, calculated as H_2SO_4 .

1.2.3 *Water content*

The water content, determined by the method WHO/M/7.R1, shall not be higher than 1 g/kg.

1.3 Packing and marking of packages

The technical phoxim concentrate shall be packed in suitable, clean containers, as specified in the order. All packages shall bear, durably and legibly marked on the container, the following:

Manufacturer's name
Technical phoxim concentrate to specification WHO/SIT/29
Batch or reference number, and date of test
Net weight of contents
Date of manufacture

and the following minimum cautionary notice:

Phoxim is an organophosphorus compound that inhibits cholinesterase. It is poisonous if swallowed.

Keep the material out of the reach of children and well away from foodstuffs and animal feed and their containers. If poisoning occurs, call a physician. Atropine and pralidoxime are specific antidotes, and artificial respiration may be needed.

2. Methods of determining chemical and physical properties

2.1 Phoxim content

2.1.1 *Outline of method*

A solution of the sample is separated by normal phase high performance liquid chromatography on silica gel. The content of active ingredient is calculated from the peak area using pure phoxim of known purity as external standard.

2.1.2 *Special apparatus*

1. *Liquid chromatograph.* The instrument should be one designed for use with stainless steel column and equipped with a 254 nm UV spectrophotometric detector and a 5 ml injection valve.
2. *Liquid chromatographic column.* The column should be a stainless steel tube 25 cm long and 4 mm internal diameter packed with LiChrosorb Si-60 (5 mm) or equivalent.

2.1.3 *Special reagents*

Phoxim analytical standard grade of known purity, better than 990 g/kg.

Mobile phase. n-hexane - tetrahydrofuran, 96 + 4 (v/v)

2.1.4 *Preparation of the calibration solution*

Weigh (to the nearest 0.1 mg) about 100 mg of phoxim standard into a 100-ml volumetric flask, dissolve in 10 ml of tetrahydrofuran, dilute to volume with n-hexane and mix.

2.1.5 *Operating conditions for high performance liquid chromatography*

The conditions given below are typical values and may have to be adjusted to obtain optimum results from a given apparatus.

Column temperature	ambient, but within $\pm 2.5^{\circ}\text{C}$.
Flow rate	2.0 ml/min ⁻¹ .
Wave length	254 nm.
Detector sensitivity	1.0 AUFS (absorbance unit full scale).
Injection volume	5 ml.
Retention time of phoxim	about 4.8 min.

2.1.6 *Sample preparation*

Weigh (to the nearest 0.1 mg) sufficient sample to contain 100 mg of phoxim into a 100 ml volumetric flask, dissolve in 10 ml of tetrahydrofuran, dilute to volume with *n*-hexane and mix.

2.1.7 *Analysis of the sample*

Pump the mobile phase through the column until the system is equilibrated. Inject 5 ml of the calibration solution and adjust operating parameters to cause phoxim to elute in about 4.8 min.

Make repetitive injections of the calibration solution and calculate the calibration factor

$$f = \frac{s \times P}{H_s}$$

where s = mass of standard phoxim in the calibration solution (mg)
 P = purity of the standard phoxim (g/kg)
 H_s = peak area of phoxim in the calibration solution.

The calibration factor (f) must agree within less than 1% for consecutive injections.

Inject 5 ml portions of the sample solution. Repeat the injection of the calibration solution after every fourth determination.

The calibration factors of the calibration solution injections immediately preceding and following sample injections must agree within less than 1%. If not, repeat the analysis.

2.1.8 *Calculation*

$$\text{Phoxim content (g/kg)} = \frac{H_w \times f}{w}$$

where f = calibration factor
 H_w = peak area of phoxim in the sample solution
 w = mass of sample taken (mg)

PHOXIM EMULSIFIABLE CONCENTRATE

Specification WHO/SIF/51
Approved 25 September 1989

1. Specification

1.1 Description and ingredients

The material shall consist of technical phoxim dissolved in suitable solvents, with other necessary formulants added. It shall be in the form of a stable liquid, free from suspended matter and sediment. The phoxim technical concentrate used in the manufacture of the emulsifiable concentrate shall comply with the requirements of specification WHO/SIT/29.

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment (see method WHO/M/1), shall comply with the requirements of section 1.1 and with the following requirements:

1.2.1 *Phoxim content (g/kg basis)*

The content of phoxim, determined by the method described in section 2.1, shall not differ from the nominal content by more than the following amounts:

<i>Nominal content</i>	<i>Tolerance permitted</i>
Up to 500 g/kg	± 5% of the nominal content
Above 500 g/kg	± 25 g/kg

The average content of all samples taken shall not be lower than the nominal content.

1.2.2 *Water content*

The water content, determined by the method described in WHO/M/7.R1, shall not be higher than 2 g/kg.

1.2.3 *Acidity*

The acidity, determined by method WHO/M/3, shall not be higher than 5 g/kg calculated as H₂SO₄. In the event of a dispute, the methyl red indicator determination shall be the referee method.

1.2.4 *Cold test*

No separation of solid or oily material shall occur when the concentrate is tested as described in method WHO/M/23.

1.2.5 *Flash point*

The flash point of the product shall comply with all national and/or international transport regulations (see method WHO/M/10.R1).

1.2.6 *Stability of the emulsion*

In standard soft water. Any separation, including creaming/oiling at the top and oiling/sedimentation at the bottom, of 100 ml of emulsion prepared in standard soft water with 5 ml of concentrate shall not exceed 2 ml when tested as described in WHO/M/13.R3.

In standard hard water. Any separation including creaming/oiling at the top and oiling/sedimentation at the bottom, of 100 ml of emulsion prepared in standard hard water with 5 ml of concentrate shall not exceed 2 ml when tested as described in WHO/M/13.R3.

1.2.7 *Heat stability*

The emulsifiable concentrate, after treatment as described in section 2.2, shall comply with the requirements of sections 1.2.1, 1.2.3 and 1.2.6 of this specification.

1.3 Packing and marking of packages

The phoxim emulsifiable concentrate shall be packed in suitable, clean containers, as specified in the order. All packages shall bear, durably and legibly marked on the container, the following:

Manufacturer's name
Phoxim emulsifiable concentrate to specification WHO/SIF/51
Phoxim g/kg
Batch or reference number, and date of test
Net weight of contents
Instructions for dilution

and the following minimum cautionary notice:

Phoxim is an organophosphorus compound that inhibits cholinesterase. It is poisonous if swallowed.

Keep the material out of the reach of children and well away from foodstuffs and animal feed and their containers. If poisoning occurs, call a physician. Atropine and pralidoxime are specific antidotes, and artificial respiration may be needed.

2. Methods of determining chemical and physical properties

2.1 Phoxim content

2.1.1 *Outline of method*

A solution of the sample is separated by normal phase high performance liquid chromatography on silica gel. The content of active ingredient is calculated from the peak area using pure phoxim of known purity as external standard.

2.1.2 *Special apparatus*

1. *Liquid chromatograph.* The instrument should be one designed for use with stainless steel column and equipped with a 254 nm UV spectrophotometric detector and a 5 ml injection valve.
2. *Liquid chromatographic column.* The column should be a stainless steel tube 25 cm long and 4 mm internal diameter packed with LiChrosorb Si-60 (5 mm) or equivalent.

2.1.3 *Special reagents*

Phoxim analytical standard grade, of known purity, better than 990 g/kg.

Mobile phase. n-hexane and tetrahydrofuran, 96 + 4 (v/v)

2.1.4 *Preparation of the calibration solution*

Weigh (to the nearest 0.1 mg) about 100 mg of phoxim standard into a 100 ml volumetric flask, dissolve in 10 ml of tetrahydrofuran, dilute to volume with n-hexane and mix.

2.1.5 *Operating conditions for high performance liquid chromatography*

The conditions given below are typical values and may have to be adjusted to obtain optimum results from a given apparatus.

Column temperature	ambient, but within $\pm 2.5^{\circ}\text{C}$.
Flow rate	2.0 ml/min ⁻¹ .
Wave length	254 nm.
Detector sensitivity	1.0 AUFS (absorbance unit full scale).
Injection volume	5 ml.
Retention time of phoxim	about 4.8 min.

2.1.6 *Sample preparation*

Weigh (to the nearest 0.1 mg) sufficient sample to contain 100 mg of phoxim into a 100 ml volumetric flask, dissolve in 10 ml of tetrahydrofuran, dilute to volume with *n*-hexane and mix.

2.1.7 *Analysis of sample*

Pump the mobile phase through the column until the system is equilibrated. Inject 5 ml of the calibration solution and adjust operating parameters to cause phoxim to elute in about 4.8 min.

Make repetitive injections of the calibration solution and calculate the calibration factor

$$f = \frac{s \times P}{H_s}$$

where s = mass of standard phoxim in the calibration solution (mg)
 P = purity of the standard phoxim (g/kg)
 H_s = peak area of phoxim in the calibration solution.

The calibration factor (f) must agree within less than 1% for consecutive injections.

Inject 5 ml portions of the sample solution. Repeat the injection of the calibration solution after every fourth determination.

The calibration factors of the calibration solution injections immediately preceding and following sample injections must agree within less than 1%. If not, repeat the analysis.

2.1.8 *Calculation*

$$\text{Phoxim content (g/kg)} = \frac{H_w \times f}{w}$$

where f = calibration factor
 H_w = peak area of phoxim in the sample solution
 w = mass of sample taken (mg)

2.2 **Heat stability**

Keep 100 ml of the sample for 3 days at a temperature of $54 \pm 2^\circ\text{C}$ in a glass container sealed to avoid loss of volatile solvent, and then cool to room temperature.