US ERA ARCHIVE DOCUMENT

DEET WHO/SRpT/1.R2

TECHNICAL DEET

Full specification WHO/SRpT/1.R2 Revised 10 December 1999

1. Specification

1.1 Description

The material shall consist of deet together with related manufacturing impurities and shall be in the form of a clear, nearly odourless liquid, free from extraneous matter or added modifying agents.

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment (see method WHO/M/1.R1), shall comply with the requirements of section 1.1 and with the following requirements.

	Minimum	Maximum
Dest content (section 2.1)	050 0/10	
Deet content (section 2.1)	950 g/kg	
Acidity (CIPAC method MT 31,		
CIPAC Handbook F, p.96) calculated as H ₂ SO ₄		0.3 g/kg
Water content (method WHO/M/7.R1)		3 g/kg
Relative density at 25 ^o C (CIPAC method MT 3.2		
CIPAC Handbook F, p.13)	0.992	0.999
Refractive index n ²⁵ _D (section 2.2)	1.520	1.524
Colour (section 2.3), Hazen number		100

1.3 Packing and marking of packages

The technical deet 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 deet Batch or reference number, and date of test WHO/SRpT/1.R2

Net weight of contents Date of manufacture

2. Methods of determining chemical and physical properties

2.1 Deet content

2.1.1 Summary of method

The sample is dissolved in carbon disulfide and the difference in absorbance at 14.18 μm and at 14.48 μm is determined. The quantity of meta-isomer is obtained from this value by means of a calibration curve prepared by the use of a reference standard.

2.1.2 Apparatus

- 1. Double-beam infrared spectrophotometer. Perkin-Elmer model 21 or equivalent.
- 2. Two equivalent infrared absorption cells, with sodium chloride windows and a path length of approximately 0.4 mm.

2.1.3 Preparation of calibration curve

Weigh (to the nearest 0.1 mg) into four volumetric flasks sufficient amounts of the reference deet standard of known purity to give concentrations of approximately 20, 40, 60, and 80 g/L when dissolved in carbon disulfide.

Fill the reference cell with carbon disulfide and the sample cell with each of the standard solutions in turn, and record the spectra. The spectrum may be scanned rapidly, except for the region 12-15 μ m, where a normal speed should be used. Carry out a blank measurement with carbon disulfide to correct for any inequality in the paired cells and to determine whether a cell correction is required.

Measure the absorbance at 14.18 μm and at 14.48 μm and calculate the difference between these values, ΔA , for each of the solutions. Plot the values of ΔA against the concentration (g/l) of the meta-isomer.

If a cell correction is required, the value of ΔA is determined from the formula:

$$\Delta A = [A_{14.18} - A_{14.48}]_{ref.} - [A_{14.48}]_{blank}$$

where ref. = determination with reference standard blank = determination on CS₂ blank.

2.1.4 *Procedure*

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Weigh (to the nearest 0.1~mg) about 0.5~g of the sample, transfer quantitatively to a 10~mL volumetric flask, and make up to the mark with carbon disulfide. Measure the infrared absorption at $14.18~\mu\text{m}$ and $14.48~\mu\text{m}$ using the same conditions as described in section 2.1.3. Determine the concentration of meta-isomer by comparing this value with the calibration curve. A standard sample should be run each day to check the calibration of the instrument.

2.1.5 Calculation

Deet content
$$(g / kg) = \frac{C_1 x P}{C_2}$$

 $C_1 = \text{concentration (g/L) of standard deet found from calibration curve}$

 C_2 = concentration (g/L) of sample taken P = purity (g/kg) of the reference standard.

2.2 Refractive index

The refractive index shall be determined by any method accurate to the third decimal place.

2.3 Colour

2.3.1 Preparation of Hazen colour standard solutions

Dissolve 1.245 g of potassium chloroplatinate (K_2PtCl_6) and 1 g of crystallized cobalt (II) chloride ($CoCl_2.6H_2O$) in 100 mL of concentrated hydrochloric acid and dilute with distilled water to make 1 litre. This standard solution has a Hazen colour number of 500. Solutions having Hazen colour numbers of 5, 10, 15, 20, 25, 30, 35, 40, 45, 50, 60, 70, 80, 90, and 100 are made by diluting in standard tall-form 500 mL Nessler tubes 0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, 5, 6, 7, 8, 9, and 10 mL of the standard solution with distilled water to make 50 mL.

2.3.2 Procedure

Place 50 mL of deet standard in a matched Nessler colour-comparison tube and match with the comparison standards (section 2.3.1). Use a suitable colorimeter provided with a white light.