

HYDROGENATED POLY-1-DECENE

Prepared at the 57th JECFA (2001) and published in FNP 52 Add 9 (2001), superseding specifications prepared at the 53rd JECFA (1999), published in FNP 52 Add 7 (1999). An ADI of 0 - 6 mg/kg bw was established at the 57th JECFA (2001).

SYNONYMS

Hydrogenated polydec-1-ene, Hydrogenated poly- α -olefin; INS No. 907

DEFINITION

Hydrogenated poly-1-decene is a mixture of branched isomeric hydrocarbons, prepared by hydrogenation of mixtures of trimers, tetramers, pentamers and hexamers of 1-decenes. Minor amounts of molecules with carbon number less than 30 may be present.

C.A.S. number

68037-01-4

Chemical formula

$C_{10n}H_{20n+2}$, where $n = 3 - 6$

Formula weight

560 (average)

Assay

Not less than 98.5% of hydrogenated poly-1-decene, having the following oligomer distribution:

C_{30} : 13 - 37%

C_{40} : 35 - 70%

C_{50} : 9 - 25%

C_{60} : 1 - 7%

DESCRIPTION

Colourless, odourless, viscous liquid

FUNCTIONAL USES

Glazing agent, release agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Insoluble in water; slightly soluble in ethanol; soluble in toluene

Burning

The product burns with bright flame and a paraffin-like characteristic smell

Viscosity

5.7 - 6.1 mm²/sec (100°)
See description under TESTS

PURITY

Compounds with carbon number less than 30

Not more than 1.5 %
See METHOD OF ASSAY

Readily carbonizable substances

Passes test
See description under TESTS

Nickel (Vol. 4)

Not more than 1 mg/kg

Lead (Vol. 4)

Not more than 1 mg/kg

Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described on Volume 4, "Instrumental methods".

TESTS

PURITY TESTS

Viscosity, 100°

Use a viscometer of the glass capillary type, calibrated and capable of measuring kinematic viscosity with a repeatability exceeding 0.35% only in one case in twenty. Immerse the viscometer in a liquid bath at the temperature required for the test $\pm 0.1^\circ$ ensuring that at no time of the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath. Charge the viscometer with sample in the manner dictated by the design of the instrument. Allow the sample to remain in the bath for about 30 min. Where the design of the viscometer requires it, adjust the volume of sample to the mark. Use pressure to adjust the head level of the sample to a position in the capillary arm of the instrument about 5 mm ahead of the first mark. With the sample flowing freely, measure, in seconds (± 0.2 sec), the time required for the meniscus to pass from the first to the second timing mark. If the time is less than 200 s, select a viscometer with a capillary of smaller diameter and repeat the operation. Make a second measurement of the flow time. If two measurements agree within 0.2%, use the average for calculating the kinematic viscosity. If the measurements do not agree, repeat the determination after thoroughly cleaning and drying the viscometer.

$$\text{Viscosity, } 100^\circ \text{ (mm}^2\text{/sec)} = C \times t$$

where

C = calibration constant of the viscometer ($\text{mm}^2\text{/sec}^2$)

t = flow time (sec)

Readily carbonizable substances

Place 5g of the sample in a glass-stoppered test tube that has previously been cleaned with a chromic acid cleaning solution, rinsed with water and dried in an oven (105° , 1h). Add 5 ml of sulfuric acid TS, and place in a boiling water bath. After the test tube has been in the bath for 30 sec, remove quickly, and while holding the stopper in place, give three vigorous vertical shakes over an amplitude of about 10 cm. Repeat every 30 sec. Do not keep the test tube out of the bath longer than 3 sec for each shaking period. At the end of 10 min from the time when first placed in the water bath, remove the test tube. The sample remains unchanged in colour, and the acid does not become darker than a very slight straw colour (Matching Fluid E, see FNP 5). No black material occurs at the interface between the two layers

METHOD OF ASSAY

Hydrogenated poly-1-decene, oligomer distribution and the content of hydrogenated decene dimer. Determine by gas-liquid chromatography (see Volume 4) using the following conditions:

Column: Capillary column, 16 m x 0.53 mm
Column film: Nonpolar (polydimethylsiloxane), thickness 0.1 μm , e.g. OV-1, DB 1
Column temperature:
- initial: 35°, 1 min
- 35° - 50°, 5°/min
- 50° - 170°, 12°/min
- 170° - 310°, 10°/min
final: 310°, 45 min
Carrier
- gas: Helium
- flow: 10 ml/min
Injector pressure: 1.5 psi at 35°, constant flow
Detector: FID
Injection temperature: 35°
Sample size: 2 μl

Calibration

Calibrate the instrument using a solution with known composition containing about 1.0 % of n-tetradecane, 1.0% of n-hexadecane and 1.0% of squalane (2,6,10,15,19,23-hexamethyl tetracosane) in pentane. The differences between peak areas of the compounds should be less than 10.0% and the resolution between tetradecane and n-hexadecane should be between 2 and 5. Calculate the resolution (R) from the distance between n-tetradecane and n-hexadecane peaks at the peak maxima (d) and the width of the peaks at the baseline (Y): $R = [2(d_1 - d_2)/(Y_1 - Y_2)]$.

Sample preparation

Take a small amount of the sample into an automatic pipette. Place a drop of the sample in 1.00 ml of pentane, and mix well. Record the chromatogram for about 45 min.

Retention times

The retention times for C_{20} , C_{30} , C_{40} , C_{50} and C_{60} are usually about 10.5, 16, 20, 23 and 26 min.

Calculation

Calculate the oligomer distribution by the method of area percentages (area normalization).

Calculate the percentage of molecules with carbon numbers less than 30 in the sample taken by the formula:

$$100 \cdot L/T$$

Where

L = the sum of peak areas eluting before and C_{30}

T = the sum of all peak areas eluting in 45 min

Calculate the percentage of hydrogenated poly-1-decene in the sample taken by the formula:

$$100 \cdot A/T$$

where

A = the sum of peak areas for C₃₀, C₄₀, C₅₀ and C₆₀

T = the sum of all peak areas eluting in 45 min