

## MINERAL OIL (MEDIUM AND LOW VISCOSITY)

*Prepared at the 59th JECFA (2002), published in FNP 52 Add 10 (2002) superseding specifications prepared at the 51st JECFA (1998), published in FNP 52 Add 6 (1998). At the 59th JECFA for mineral oils of Class I an ADI of 0-10 mg/kg bw and for minerals oils of Class II and Class III a temporary group ADI of 0-0.01 mg/kg bw were established.*

<b>SYNONYMS</b>	Liquid paraffin, liquid petrolatum, food grade mineral oil, white mineral oil INS No. 905a
<b>DEFINITION</b>	A mixture of highly refined paraffinic and naphthenic liquid hydrocarbons with boiling point above 200°; obtained from mineral crude oils through various refining steps (eg. distillation, extraction and crystallisation) and subsequent purification by acid and/or catalytic hydrotreatment; may contain antioxidants approved for food use. The oil is divided into three classes having different viscosities and average molecular weights as specified below.
C.A.S. number	8012-95-1
<b>DESCRIPTION</b>	Colourless, transparent, oily liquid, free from fluorescence in daylight; odourless
<b>FUNCTIONAL USES</b>	Release agent, glazing agent
<b>CHARACTERISTICS</b>	
IDENTIFICATION	
<u>Solubility</u> (Vol. 4)	Insoluble in water, sparingly soluble in ethanol, soluble in ether
<u>Burning</u>	Burns with bright flame and with paraffin-like characteristic smell
PURITY	
<u>Viscosity, 100°</u>	Class I: 8.5-11 mm <sup>2</sup> /s Class II: 7.0-8.5 mm <sup>2</sup> /s Class III: 3.0-7.0 mm <sup>2</sup> /s See description under TESTS
<u>Carbon number at 5% distillation point</u>	Class I: Not less than 25 Class II: Not less than 22 Class III: Not less than 17 The boiling point at the 5% distillation point is higher than: 391° for Class I substances, 356° for Class II substances and 287° for Class III substances. See description under TESTS
<u>Average molecular weight</u>	Class I: 480-500 Class II: 400-480 Class III: 300-400 See description under TESTS

Acidity or alkalinity To 10 ml of the sample add 20 ml of boiling water and shake vigorously for 1 min. Separate the aqueous layer and filter. To 10 ml of the filtrate, add 0.1 ml of phenolphthalein solution TS. The solution is colourless. Not more than 0.1 ml of 0.1N sodium hydroxide is required to change the colour to pink

Readily carbonizable substances Place 5 ml of the sample in a glass-stoppered test tube that has previously been rinsed with chromic acid cleaning mixture. Add 5 ml of sulfuric acid TS, and heat in a boiling water bath for 10 min. After the test tube has been in the bath for 30 sec, remove it 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 standard colour produced by mixing in a similar test tube 3 ml of ferric chloride TSC, 1.5 ml of cobaltous chloride TSC, and 0.5 ml of cupric sulfate TSC, this mixture being overlaid with 5 ml of mineral oil.

Polycyclic aromatic hydrocarbons Transfer 25.0 ml of sample to a 125 ml separating funnel with unlubricated ground-glass parts (stopper, stopcock). Add 25 ml of hexane which has been previously shaken twice with one-fifth its volume of dimethyl sulfoxide. Mix and add 5.0 ml of dimethyl sulfoxide. Shake vigorously for 1 min and allow to stand until two clear layers are formed. Transfer the lower layer to a second separating funnel, add 2 ml of hexane and shake the mixture vigorously. Allow to stand until two clear layers are formed. Separate the lower layer and measure its absorbance between 260 nm and 420 nm, using as the compensation liquid the clear lower layer obtained by vigorously shaking 5.0 ml of dimethyl sulfoxide with 25 ml of hexane for 1 min. Prepare a reference solution in trimethylpentane counting 7.0 mg of naphthalene per litre and measure the absorbance of the solution at the maximum at 275 nm, using trimethylpentane as the compensation liquid. At no wavelength between 260 nm and 420 nm does the absorbance of the test solution exceed one-third that of the reference solution at 275 nm. Use hexane, dimethyl sulfoxide and trimethylpentane in quality specified for ultraviolet spectrometry.

Solid paraffins Dry a suitable quantity of the substance to be examined by heating at 100° for 2 h and cool in a desiccator over concentrated sulfuric acid. Place in a glass tube with an internal diameter of about 25 mm, close the tube and immerse in a bath of iced water. After 4 h the liquid is sufficiently clear for a black line, 0.5 mm wide against a white background held vertically behind the tube, to be easily seen.

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 the method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods".

## TESTS

## PURITY TESTS

### Viscosity, 100°

ASTM D 445

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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 ( $\pm 0.2$  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.

Viscosity, 100° (mm<sup>2</sup>/sec) = C x t

Where

C = calibration constant of the viscometer (mm<sup>2</sup>/sec<sup>2</sup>)

t = flow time (sec)

### Carbon number

ASTM D 2887

See TEST for Viscosity for  
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"Carbon number" is number of carbon atoms in a molecule. Determine the boiling point distribution of the sample by *gas chromatography* using the following conditions:

The system must have the following performance characteristics:

#### Sensitivity

: 1% dodecane must be detected with a peak height of at least 10% of full scale under the conditions prescribed below.

#### Stability

: when operated at the required sensitivity level, the baseline drift is not more than 1% of full scale per hour

#### Repeatability of retention times

: 6 sec for each component of the calibration mixture.

#### Resolution (R)

: determined for a solution of 1% of each of hexadecane and octadecane in n-octane is not less than three and not more than eight, using the following formula:

$$R = \frac{2d}{W1+W2}$$

where

$d$  = distance in mm between the peak maxima of hexadecane and octadecane

$W_1$  = the peak width in mm at the baseline of hexadecane

$W_2$  = the peak width in mm at the baseline of octadecane

Typical conditions which may be used:

#### Column

length: 1.5m

outside diameter: 3.2 mm

liquid phase: SE - 30.5 %

support material: Chromosorb G, mesh 60/80

#### Column temperature

: initial: 10°

final: 350°

rate: 6.5° / min.

#### Carrier

: gas: helium

flow: 30 ml/min.

#### Detector

: FID

Detector temperature: 370°

#### Injection temperature

: 370°

#### Sample size

: 0.3  $\mu$ l

#### Calibration mixture

: Prepare a mixture of hydrocarbons of known boiling points covering the range of the sample. At least one compound must have a boiling point lower than the initial boiling point of the sample.

#### Procedure

: Calibration: Cool the column to the selected starting temperature (the retention time for the initial boiling point must be at least 1 min) and inject the calibration mixture. Record the retention time of each peak maximum and the peak areas for each component. Plot the retention time of each peak versus the corresponding normal boiling point of that component in degrees Celsius to obtain a calibration curve.

#### Sample analysis

: Using the exact conditions used in the calibration run, inject the sample. Record the area of each time segment at fixed time intervals not greater than 1% of the retention time equivalent to a boiling point of 538° obtained from the calibration curve.

#### Calculation

: Sum the area segments to obtain the cumulative area at each time

interval during the run. At the point of the chromatogram, where the baseline at the end first becomes steady, observe the cumulative area counts. Move back along the record until a cumulative area equal to 99.5% of the total at the steady point appears. Mark this point as the final boiling point. Observe the area counts at the start of the run until the point is reached, where the cumulative area count is equal to 0.5% of the total area. Mark this point as the initial boiling point of the sample. Divide the cumulative area at each interval between the initial and final boiling points by the total cumulative area and multiply by 100. This will give the cumulative percent of the sample recovered at each time interval. Tabulate the cumulative percent recovered at each interval and the retention time at the end of the interval. Using linear interpolation, if necessary, determine the retention time associated with 5% and read the corresponding boiling temperature from the calibration curve. The boiling point at the 5% distillation point is higher than: 391° for Class I substances, 356° for Class II substances and 287° for Class III substances.

Average molecular weight

ASTM D 2502  
See TEST for Viscosity for  
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Determine the kinematic viscosity of the sample at 37.8 and 98.9° as described in the method for Viscosity, 100°. Read the value of H that corresponds to the measured viscosity at 37.8° by the use of table 1; linear interpolation between adjacent columns may be required. Read a viscosity -molecular weight chart for H and 98.9° viscosity (the chart is available from the American Society for Testing and Materials (ASTM). A simplified version is shown in Figure 1 for illustration purposes only. Interpolate where necessary between adjacent lines of 98.89° viscosity. After locating the point corresponding to the value of H (ordinate) and the 98.89° viscosity (superimposed lines), read the molecular weight along the abscissa.

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.

Table 1 - Tabulation of H Function					
Kinematic viscosity, mm <sup>2</sup> /s at 37.8°	H				
	0	0.2	0.4	0.6	0.8
2	-176	-151	-126	-104	-85
3	-87	-52	-38	-25	-13
4	-1	9	19	28	36
5	44	52	59	66	73
6	79	85	90	96	101
7	100	111	116	120	124
8	128	132	136	140	144
9	147	151	154	157	160
10	163	166	169	172	175
11	178	180	183	185	188
12	190	192	195	197	199
13	201	203	206	208	210
14	211	213	215	217	219
15	212	222	224	226	227
16	220	231	232	234	235
17	237	238	240	241	243
18	244	245	247	248	249
19	2551	252	253	255	258
20	257	258	259	261	262
21	263	264	265	266	267
22	269	270	271	272	273
23	274	275	276	277	278
24	279	280	281	281	282
25	283	284	285	286	287
26	288	289	289	290	291
27	292	293	294	294	295
28	296	297	298	298	299
29	300	301	301	302	303
30	304	304	305	306	306
31	307	308	308	309	310
32	310	311	312	312	313
33	314	314	315	315	316
34	317	317	318	319	319
35	320	320	321	322	322
36	323	323	324	325	325
37	325	326	327	327	328
38	328	329	329	330	331
39	331	332	332	333	333

