

MINERAL OIL (MEDIUM VISCOSITY)

Prepared at the 76th JECFA, published in FAO JECFA Monographs 13 (2012), superseding specifications for Mineral oil (Medium and low viscosity), class I prepared at the 59th JECFA (2002), published in FNP 52 Add 10 (2002) and republished in FAO JECFA Monographs 1 (2005). An ADI of 0-10 mg/kg bw was established at the 59th JECFA for mineral oil (medium and low), class I.

At the 76th JECFA the temporary ADI and the specifications for mineral oils (Medium and low viscosity), class II and class III were withdrawn.

SYNONYMS

Liquid paraffin, liquid petrolatum, food grade mineral oil, white mineral oil, INS No. 905e

DEFINITION

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.

C.A.S. number

8012-95-1

DESCRIPTION

Colourless, transparent, oily liquid, free from fluorescence in daylight; odourless

FUNCTIONAL USES

Release agent, glazing agent

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Insoluble in water, sparingly soluble in ethanol, soluble in ether

Burning

Burns with bright flame and with paraffin-like characteristic smell

PURITY

Viscosity, 100°

8.5-11 mm²/s
See description under TESTS

Carbon number at 5%
distillation point

Not less than 25
The boiling point at the 5% distillation point is higher than: 391°.
See description under TESTS

Average molecular
weight

480-500
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 containing 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 AAS (Electrothermal atomization technique) appropriate to the specified level. The selection of sample size and method of sample preparation may be based on principles of methods

described in Volume 4 (under "General Methods, Metallic Impurities").

TESTS

PURITY TESTS

Viscosity, 100°

ASTM D 445
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from the Annual Book of
ASTM Standards,
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Society for Testing and
Materials, 100 Harbor
Drive, West
Conshohocken, PA 19428.

Copies of the complete
ASTM standard may be
purchased directly from
ASTM, phone: +1 610-
832-9585, fax: +1 610-
832-9555
e-mail: service@astm.org
<http://www.astm.org>

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)

Carbon number

ASTM D 2887
See TEST for Viscosity
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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 is distance in mm between the peak maxima of hexadecane and octadecane

W1 is the peak width in mm at the baseline of hexadecane

W2 is 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 µl

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°.

Average molecular weight
ASTM D 2502
See TEST for Viscosity
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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.

Kinematic viscosity, mm ² /ls at 37.8°	H									
	0	0.2	0.4	0.6	0.8					
2	-176	-151	-126	-104	-85					
3	-67	-52	-38	-25	-13					
4	-1	9	19	28	36					
5	44	52	59	66	73					
6	79	85	90	96	101					
7	106	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	229	231	232	234	235					
17	237	238	240	241	243					
18	244	245	247	248	249					
19	2551	252	253	255	256					
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					
38	323	323	324	325	325					
37	325	326	327	327	328					
38	328	329	329	330	331					
39	331	332	332	333	333					
	H									
	0	1	2	3	4	5	6	8	9	10
40	334	336	339	341	343	345	347	349	352	354
50	355	357	359	361	363	364	366	368	369	371
60	371	374	375	377	378	380	381	382	384	385
70	386	387	388	390	391	392	393	394	395	397

80	398	399	400	401	402	403	404	405	406	407
90	409	409	410	410	411	412	413	414	415	416
100	416	417	418	419	420	420	421	422	423	423
110	424	425	425	426	427	428	428	429	430	430
120	431	432	432	433	433	434	435	435	436	437
130	437	438	438	439	439	440	441	441	442	442
140	443	443	444	444	445	446	446	447	447	448
150	448	449	449	450	450	450	451	451	452	452
160	453	453	454	454	455	455	456	456	456	457
170	457	458	458	459	459	460	460	460	461	461
180	461	462	462	463	463	463	464	464	465	465
190	465	466	466	466	467	467	468	468	468	469
	H									
	0	10	20	30	40	50	60	10	80	90
200	469	473	476	479	482	485	487	490	492	495
300	497	499	501	503	505	507	509	511	512	514
400	515	517	518	520	521	523	524	525	527	528
500	529	530	531	533	534	535	536	537	538	539
600	540	541	542	543	544	545	546	547	547	548
700	549	550	551	551	552	553	554	554	555	556
800	557	557	558	559	559	560	561	562	562	563
900	563	564	565	565	566	566	567	567	568	569
	H									
	0	100	200	300	400	500	600	700	800	900
1000	569	574	578	583	587	591	594	597	600	603
2000	605	608	610	614	616	618	620	621	623	625
3000	625	626	628	629	631	632	633	634	636	637
4000	638	639	640	641	642	643	644	645	646	647
5000	648	649	650	651	652	652	653	654	655	656
6000	656	657	658	658	659	660	660	661	662	662
7000	663	664	664	665	665	666	666	667	667	668
8000	668	669	670	670	671	671	671	672	672	673
9000	673	674	674	675	675	676	676	677	677	677
	H									
	0	1000	2000	3000	4000	5000	6000	7000	8000	9000
10000	678	681	684	688	691	694	696	699	701	703
20000	705	707	709	711	712	715	715	717	718	719
30000	720	722	723	724	725	726	727	728	729	730
40000	731	732	732	733	734	735	736	736	737	738
50000	739	739	740	741	741	742	743	743	744	744
60000	745	746	746	747	747	748	748	749	749	750
70000	750	751	751	752	752	753	753	753	754	754
80000	755	755	756	756	756	757	757	758	758	758
90000	759	759	759	760	760	760	761	761	761	762
100000	762	762	763	763	763	764	764	764	764	765

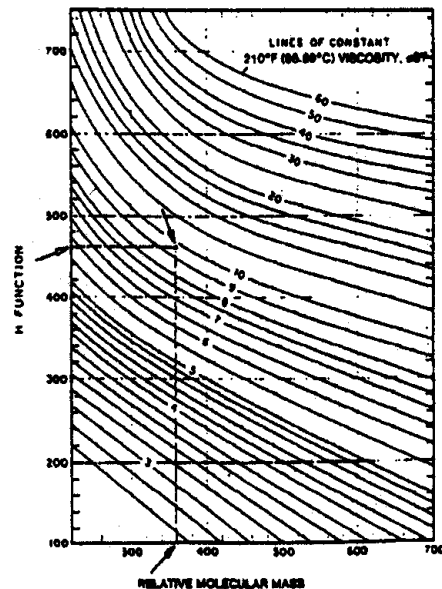


Figure 1 – Lines of constant viscosity (mm^2/s) at 98.89°

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 [Volume 4](#)). No black material occurs at the interface between the two layers.