

JIS

JAPANESE INDUSTRIAL STANDARD

**Crude petroleum and
petroleum products — Sampling**

JIS K 2251—1991

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In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

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JAPANESE INDUSTRIAL STANDARD

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Crude petroleum and petroleum products
- Sampling

K 2251-1991

1. Scope

This Japanese Industrial Standard specifies the sampling method of representative sample for testing the qualities of crude petroleum and petroleum products from inventory, loads, etc. However, except liquefied petroleum gases, electrical insulating oils and aromatic products.

Remarks 1. The sampling methods of samples of vapor-pressure tests for crude petroleum, automobile gasoline, aviation gasoline, aviation turbine fuel are shown in Annex (Sampling method for samples for vapor pressure tests).

2. Applicable Standards to this Standard are shown in the following.

JIS C 3105-Hard-Drawn Copper Stranded Conductors

JIS K 2220-Lubricating Grease

JIS K 2258-Testing Method for Vapor Pressure of Crude Oil
and Petroleum Products (Reid Method)

JIS K 2839-Glasswares for Testing Apparatus of Petroleum
Products

JIS L 2701-Manila and Sisal Fibre Ropes

JIS L 2702-Cotton Rope

JIS M 8100-General Rules for Methods of Sampling of Bulk
Materials

JIS Z 8101-Glossary of Terms Used in Quality Control

JIS Z 9001-General Rules for Sampling Inspection Procedures

3. The corresponding International Standard to this Standard is shown in the following.

ISO 3170-1988 Petroleum liquids-Manual sampling

4. In this Standard the units and numerical values shown in { } are based on the traditional units and are currently the criteria in force.

Further, in this Standard the traditional units and numerical values shown in { } are to become the informative reference starting April 1, 1995.

2. General matters

General matters used in this Standard are as follows.

- (1) Terms common to quality control are in accordance with JIS Z 8101.
- (2) General matters relating to sampling inspection are in accordance with JIS Z 9001.

3. Definitions

For the purposes of this Standard the following definitions apply:

- (1) lot The population of crude petroleum or petroleum products for sampling representative sample.

Remarks: In case where the manufacturing lot can not specify particularly such as in receiving station where it is preferable to make the manufacturing lot as it is the lot, the collection of delivery products at one time may be made the lot.

- (a) stationary lot The lot in the stationary condition such as inventory in the same tank, goods shipped from the same tank, goods stored under the equal conditions, etc. For example, it means the tank, ship, lorry, drum lot.
- (b) moving lot The lot of goods produced under the equal conditions or to be considered to be produced which are under the continuous moving condition. For example, it means apparatus discharging line, blender line, pipe line.
- (2) primary sample The generic name of samples taken directly from the lot and the primary sample means the following.
 - (a) all-layer sample Sink a sampler with weight or porcelain sampler applied with plug as it is to the height of outlet line of tank bottom part as far as possible, then take the plug and while drawing up at approximately a constant speed so as the sampler to be filled by approximately 3/4 until completion of drawing up, take the sample⁽¹⁾ from the tank.

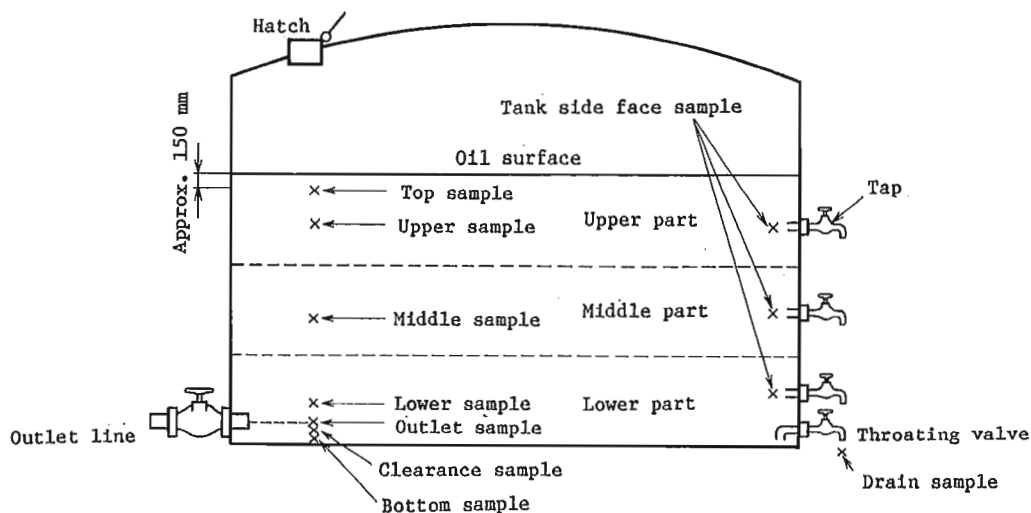
Note ⁽¹⁾ For taking the all-layer sample from a barrel or drum, use the fine tube sampling method.

Informative reference: Because the entering speed of sample into the sampler is proportional to square root of oil depth, by drawing up the sampler at a constant speed, the sample proportional to oil quantity of each part cannot be taken. Therefore, the sample taken by this method is not proportional to oil quantity of each part.

- (b) running sample After sinking a sample with a weight or porcelain sampler with a plug detached as it is at an approximately constant speed to the height of the outlet line of the bottom part of the tank, take the sample so as the sampler to be filled approximately 3/4 from the tank by the time of the drawing up to the oil surface.

- (c) spot sample A sample taken by using a sampler with a weight, porcelain sampler, cylinder sampler or the like from a specific location in the tank or the sample taken from the pipe line during transferring of lot at specific timing.
- (d) upper sample A sample taken from the middle part at the upper part $1/3$ of oil depth in the tank (refer to Fig. 1).
- (e) middle sample A sample taken from the middle part of the middle part $1/3$ of oil depth in the tank (refer to Fig. 1).
- (f) lower sample A sample taken from the middle part of lower part $1/3$ of oil depth in the tank (refer to Fig. 1).
- (g) initial-period sample A sample taken from the initial $1/6$ of total transmitting quantity of pipe line during transferring of lot.
- (h) medium-period sample A sample taken from the total transfer quantity $1/2$ of pipe line during transferring of lot.
- (i) after-period sample A sample taken from the total transfer quantity $5/6$ of pipe line during transferring of lot.
- (j) circulating line sample A sample taken from the sampling opening provided at the circulating system for making the crude petroleum and petroleum product in the tank uniform as the representative sample to be able to be obtained.
- (k) continuous sample A sample taken continuously from the pipe line during transferring of lot so as to be able to obtain the representative sample.
- (l) dipping-up sample A sample taking a specific quantity at each specific time when the flow rate is constant, by receiving the exposing-out flow with dipping up device or other collecting device, or in the case of not specific flow rate, corresponding to variation of flow-rate variation at different time intervals, the sample taken by specific quantity from all lateral sections of respective flows.

Fig. 1. Position of sampling in a fixed tank
(An example)



- (3) Secondary sample The generic name of samples which are prepared by mixing equal quantity or equal ratio or the like of primary sample to be used for test or the like as the representative sample of lot, and the secondary sample means the following.

Informative reference: The primary sample as it is made the secondary sample in some cases.

- (a) mean sample The sample mixed with samples taken from each part of tank or the like at the ratio of each part quantity.
- (b) mixed sample The sample mixed-in with equal quantities of spot samples at two or more locations sampled from the same tank. This term applies also to the sample taken from the pipeline.
- (c) single-tank mixed sample The sample mixed with samples of each part in the same tank. In the case of a tank having a uniform lateral section such as vertical cylinder type tank, it means the sample mixed with equal quantity of each part. In the case of a horizontal cylinder type tank, it means the sample mixed with the parts taken as given in Table 2.
- (d) multi-tank all-layer mixed sample The sample mixed at the ratio of oil quantity of each tank or each hatch by taking the all-layer sample from each hatch of tank, ship, barge tank, etc. containing the crude petroleum and petroleum products of the same kind.
- (e) multi-tank spot mixed sample The sample mixed in equal ratio, corresponding to oil quantities of respective tanks or hatches, by taking spot samples of several types from each part of many tanks, ships, barge tanks, etc.

- (4) local sample The generic name of samples taken from limited parts (locations) of tank, drum, etc. and the local sample means the following.
- (a) top sample The sample taken from the portion of approximately 150 mm under the oil surface of tank (refer to Fig. 1).
 - (b) outlet sample The sample taken from outlet line or swing line face of tank (refer to Fig. 1).
 - (c) clearance sample The sample taken from the portion of approximately 100 mm under the outlet line of tank (refer to Fig. 1).
 - (d) bottom sample The sample taken from the lowest part of bottom of the tank, drum, can or pipe line (refer to Fig. 1).
 - (e) drain sample The sample taken from the throating valve of tank (refer to Fig. 1) or from the exhaust valve of tank car.

Informative reference: In the case of tank car, in some cases it is the same as the bottom sample.

- (f) bottom-sediment-and-water sample The sample taken from sediments at the bottom part of tank and the like by means of sheaf or the like.

4. Primary sampling method in type division of lot

4.1 Sampling method of primary sample and name of secondary sample Take out the primary sample from crude petroleum and petroleum product, and prepare secondary sample from this primary sample. These samples are used for testing of crude petroleum and petroleum products and therefore these shall be so taken that these have mean characteristics of quality. The example of sampling method of primary sample and name of secondary sample name are shown in Table 1.

Table 1. Sampling method of primary sample and names of secondary samples (Example)

Type of lot		Primary sample			Name of secondary sample
		Name of sample	Specification clause number of sampling pieces	Name of sampling method	
Stationary lot	Fixed tank (vertical cylinder tank, horizontal cylinder tank, etc.)	Upper sample	4.2.2 (2)	Sampling method by sampler with weight	Mean sample Mixed sample Single tank mixed sample Multi-tank spot mixed sample
		Middle sample		Tap sampling method	
		Lower sample			
		All-layer sample	-	Sampling method by sampler with weight	All-layer sample

Table 1. (Continued)

Type of lot		Primary sample			Name of secondary sample
		Name of sample	Specification clause number of sampling pieces	Name of sampling method	
		Circulating line sample	-	Circulating line sampling method	Circulating line sample
	Moving tank (tank car, lorry, ship, etc.)	Upper sample	4.2.3	Sampling method by sampler with weight	Multi-tank spot mixed sample
		Middle sample			
		Lower sample			
		All-layer sample			Multi-tank all-layer mixed sample
		Continuous sample		Continuous line sampling method	Continuous sample
	Load making product (drum, barrel, can, etc.)	Spot sample	4.2.4	Siphon sampling method	Spot sample Mean sample All-layer sample
		All-layer sample		Fine tube sampling method	
Moving lot	Line (Apparatus discharge line, blender line, pipe line etc.)	First sample	-	Continuous line sampling method	Mixed sample
		Middle sample			continuous sample
		Latter sample			
		Continuous sample			Continuous sample (automatic sampling)

Note (2) In the case where in a vertical cylinder type tank of shallow petroleum depth, sampling of each part sample of upper, middle, and lower samples are impossible, the spot sample may be taken according to 4.2.1.

4.2 Selection of sampling number of pieces

4.2.1 In the case of taking spot sample from hatch of vertical cylinder type tank, ship, barge tank, etc., the sampling shall be in accordance with the following.

- (1) Three point method In a tank or the hold of a ship exceeding 5 m in petroleum depth, take from each part of upper, middle: and lower parts.
- (2) Two point method In a tank or the hold of a ship exceeding 3 to 5 m in petroleum depth, take from each part of upper and lower parts.
- (3) Single point method In a tank or the hold of a ship less than 3 m in petroleum depth, take from as near middle part of petroleum depth as possible.

4.2.2 Sampling position of primary sample from horizontal cylinder type tank and sample mixing ratio Relative to the petroleum depth, take sample and mix as shown in Table 2.

Table 2. Sampling position of horizontal cylinder type tank and sample mixing ratio

Petroleum depth (% to diameter)	Sampling position (distance from the bottom part to diameter %)			Sample mixing ratio (volume)		
	Upper	Middle	Lower	Upper	Middle	Lower
100	80	50	20	3	4	3
90	75	50	20	3	4	3
80	70	50	20	2	5	3
70		50	20		6	4
60		50	20		5	5
50		40	20		4	6
40			20			10
30			15			10
20			10			10
10			5			10

4.2.3 Number of sampling pieces of primary sample from multi-ship holds

In the case of sampling from the holds of ships, take all-layer sample, running sample or middle sample from each hold of ship according to the following reference. However, as required, the sample other than these may be sampled.

Number of holds of ships of
one type

Minimum number of holds of
ships of sampling

1 to 2 holds of ships

Each hold of ship

3 to 6 holds of ships

2 holds of ships

7 holds of ships or more

3 holds of ships

4.2.4 Number of sampling pieces of primary sample from packing

products Take packing products (can, drum, barrel or box) from individual packing container, and prepare one piece of secondary sample to represent the lot or packing products. In this case, take the individual packing container for sampling at random.

In the case where the lot number is indicated clearly, decide the number of sampling pieces according to Table 3, and in the case where the lot number is not clear, decide according to Table 4.

Table 3. Number of sampling pieces to number of packing containers
(In the case where the lot number is indicated clearly)

Number of packing containers	Number of sampling pieces	Number of packing containers	Number of sampling pieces
1 to 3	Total number	1332 to 1728	12
4 to 64	4	1729 to 2197	13
65 to 125	5	2198 to 2744	14
126 to 216	6	2745 to 3375	15
217 to 343	7	3376 to 4096	16
344 to 512	8	4097 to 4913	17
513 to 729	9	4914 to 5832	18
730 to 1000	10	5833 to 6859	19
1001 to 1331	11	6860 min.	20

Informative reference: The numbers of sampling pieces of number of packing containers of 64 or more are based on the cubic root (round-up integer) of number of packing containers.

Table 4. Number of sampling pieces to number of packing containers
(In the case where the lot number is not clear)

Number of packing containers	Number of sampling pieces	Number of packing containers	Number of sampling pieces	Number of packing containers	Number of sampling pieces
1	1	257 to 289	17	1025 to 1089	33
2 to 4	2	290 to 324	18	1090 to 1156	34
5 to 9	3	325 to 361	19	1157 to 1225	35
10 to 16	4	362 to 400	20	1226 to 1296	36
17 to 25	5	401 to 441	21	1297 to 1369	37
26 to 36	6	442 to 484	22	1370 to 1444	38
37 to 49	7	485 to 529	23	1445 to 1521	39
50 to 64	8	530 to 576	24	1522 to 1600	40
65 to 81	9	577 to 625	25	1601 to 1681	41
82 to 100	10	626 to 676	26	1682 to 1764	42
101 to 121	11	677 to 729	27	1765 to 1849	43
122 to 144	12	730 to 784	28	1850 to 1936	44
145 to 169	13	785 to 841	29	1937 to 2025	45
170 to 196	14	842 to 900	30	2026 to 2116	46
197 to 225	15	901 to 961	31	2117 to 2209	47
226 to 256	16	962 to 1024	32	2210 to 2304	48

Informative reference: The numbers of sampling pieces in this Table are based the square root (rounded up to integer) of numbers of packing containers.

5. Preparation and marking of secondary sample

5.1 Preparation of secondary sample The secondary sample shall be taken, as a rule, from the primary sample and the mixing, be carried out in the test room not at the sampling site.

- (1) In the case of preparing a secondary sample from the primary sample
In the case where the quantity of primary sample is sufficient and there is no fear of variation of its property due to transfer, mixing by stirring, mixing, etc., mix by stirring the one piece of primary sample, and mix to make uniform. Thereafter take two pieces of secondary sample and preserve the one piece. At this time, do not mix-in the bubbles in the sample, dust, and other foreign matter due to stirring, mixing, etc. In the case where for taking the secondary sample, dissolution by heating of primary sample is required, do not generate variation of property by heating.

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- (2) In the case of preparing from multi-tank spot samples Take out the spot sample according to 4.2.1 by one of three point method, two point method or single point method mix in equal ratio corresponding to respective petroleum quantities in respective tanks, and take it as a multitank spot sample.
- (3) In the case of making the primary sample as it is the secondary sample In the case where due to small capacity of sample the sampling quantity of once is small and two pieces of secondary sample can not be taken from one piece of primary sample (sampling methods by sampler with weight, porcelain sampler, cylinder sampler, fine tube sampling method, sheaf sampling method, etc.) and in the case where there is a fear for variation of sample property due to transfer, stirring, mixing, etc. (evaporating property sample, grease sample, etc.) take out two pieces of primary sample from the same lot and take them as they are as the secondary sample.

Remarks: In the case where the secondary sample is sent to user, purchaser, etc., prepare three pieces.

5.2 Marking of sample name and the like After preparing of secondary sample, attach the label entered with the following matters:

- (1) Name and type of sample
- (2) Lot number, sample number or reference number
- (3) Year, month, day, time (in the case of continuous sample, the time required for sampling) and place for sampling
- (4) Name of sampling person
- (5) Names and numbers of ship, wagon, and product containers, and manufacturer's name.
- (6) Name of hazardous matter (First petroleum, second petroleum, etc.) specified in the Fire Service Law. However, except the case of feeding to test room in the same site.

6. Caution matters for sampler and sampling

6.1 Sample container The sample container shall be as follows. However, in the case where the sample container is specified according to the test method such as water separation index, electric conductivity etc. of aviation turbine, follow each testing method.

- (1) Body of sample container
 - (a) Transparent glass bottle and dark brown glass bottle Use the dark brown bottle for matter of which property changes by light, and use the transparent glass bottle for matter of which property does not change. However, the glass bottle shall not be used for matter exceeding 180 kPa{1.84 kgf/cm²} in Reid vapor pressure.

Remarks: In case where the Reid vapor pressure of sample exceeds 110 kPa{1.12 kgf/cm²}, store the glass bottle into a metal container for safety.

- (b) Polyethylene bottle The polyethylene bottle has gas permeability and dependent upon the sample material component may elute, and therefore shall not be used for high-vapor-pressure sample and the sample of strong dissolution power in material component.
- (c) Can For the can to be used for sample in which adhesives at seam elute such as gasoline, kerosene, etc., use the can of which seams are applied with soldering, brazing or electric welding. However, the can shall be so made that the electric characteristics, oxidation stability, generating quantity of sludge are not given with influence by remaining flux.

Remarks 1. Even the can of which seams are used of adhesives, if it does not give influence the test results, it may be used.

2. As to the container to be used for transport the taken sample to other place, refer to "Hazardous Materials Control Rule" of the Fire Service Law.

- (2) Cover of sample For glass bottle, cork plug, glass plug or plastic cover shall be used. The cork plug shall be clean and fine. For can the metal cover⁽³⁾ is used, but in order to maintain the gas tightness, the internal cover shall be used together.

Note ⁽³⁾ The metal cover entered in its inside with tin foil, aluminium foil or material of quality not to act with sample as the packing is to be used.

- (3) Washing As to sample container prior to use, wash with solvent, detergent, tap water, and at last as required pure water or distilled water in this sequence, thereafter blow clean warm air in the sample container or dry in an oven and thereafter apply the cover. On the dried sample container, water content, dust, detergent, flux used for soldering or the like, acid, rust, oil shall not remain.

6.2 Cautions relating to sampling Cautions relating to sampling shall be as follows.

- (1) As a rule, carry out the sampling work bare handed, and keep the hands clean always without fail. In cases of a cold or high temperature sample, and for others in the case of requiring gloves inevitably for safety, use clean gloves. For cloth to wipe off the sample adhered on the outside of sample container, use the cloth with as little fluffing as possible so the sample will not to be stained.
- (2) In the case of sampling from tap, valves of continuous line, circulating line, etc., gradually open the valve.

Remarks: For sampling from high pressure line, tap, etc., with taking care so as the sample not to jet out rapidly, open the valve slowly.

Further, even in the case where the valve is blocked and the sample does not come out, striking the blocked matter by bar or the like is hazardous and therefore shall be avoided.

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- (3) Because many petroleum vapors are hazardous and have flammability, avoid the suction of vapor as far as possible and at the same time avoid necessarily an open flame, spark discharge of static electricity, spark due to a metallic device. Because the shoes applied with rivets made of iron have a fear for generating sparks, do not use.
- (4) After washing preliminarily the sampler and sample container with the sample to be taken as required together, use them.
- (5) In the case of transferring the comparatively high evaporating sample [the sample of 13.7 kPa (0.14 kgf/cm²) or more in Reid vapor pressure] to sample container from sampler, carry out with taking care so as not to touch the atmospheric air as far as possible.
- (6) The sampling working person, when sampling from hatch and the like, must carry out the work at a position parallel to the wind direction.

- Remarks 1. The position parallel to the wind direction means that relative to the line connecting the centers of sampling person and hatch the wind direction intersects vertically.
2. The sampling work of hazardous materials (First to fourth petroleum) instructed by the Fire Service Law is the fact that the "handling person of hazardous materials" carries out the work directly. At this time, carry out the work by two or more persons as far as possible in order to prevent the hazard.

6.3 Cautions relating to prevention of static electricity hazard The static electricity hazard at the time of sampling of petroleum products is caused when the conditions such as charging of static electricity, formation of explosive mixed gas, discharging, etc. overlap. Therefore, generally the petroleum products charge easily, but specially the aviation turbine fuel, heavy industrial gasoline, etc. easily form explosive mixed gas at ordinary temperature, and therefore at the time of sampling, care shall be taken on the following matters.

- (1) In case where lightning generation is anticipated, do not carry out the sampling work. At the time of strong winds, when wind blows in from the hatch, even the vapor concentration in the tank is at the upper limit of explosive range or more, it becomes in some cases locally the vapor concentration in the explosive range and therefore carry out carefully.
- (2) In the case where the sample in the container such as a tank is sampled from the hatch, after transfer or completion of filling, it is preferable to provide standing still time of 30 min or more.

Remarks: In the case of floating roof tank or in the case where in other tank the static electricity preventive agent is added to the petroleum product and its electric conductivity is 50 pS/m or more, the electric charge is almost not formed and therefore the standing-still time of 30 min may be reduced.

- (3) Immediately before sampling work, grip the earth bar with the bare hand or carry out removing of personal body electricity by means of removing electricity gate or the like.

Remarks: The work floor should preferably be applied with conductive painting or placed-on with conductive mat or the like.

- (4) For worker's clothing, use work clothes, shoes, etc. having good electric conductivity and during sampling work do not change clothes.

Further, as a rule, do not use gloves. However, in the case where protective gloves are required, use conductive gloves or earth the arm, band, etc. to avoid electrification of the human body.

- (5) In the case where transferring from sampler to metallic sample container is carried out at the site, also ground the sample container.
- (6) In the case where sampler and sample container are washed together, put together with the washing petroleum into another container, and after completion of sampling work, otherwise treat safely.

6.4 Cautions relating to sample handling Cautions relating to sample handling shall be as follows:

- (1) Volatile sample As to crude petroleum and volatile samples, caution must be taken to prevent evaporation loss as far as possible. Except the case where the sampler as it is used as sample container, immediately transfer the sample from sampler to sample container, always close tightly and place in a cool, dark place.

Further, in the case when the plug of the container containing the sample is opened, preliminarily the content must be cooled sufficiently.

- (2) Photo-sensitive sample Because the sample containing much unsaturated contents varies easily in quality due to light or heat, in the case where it is used for tests of color, additives content rate, sludge generating characteristics, oxidation stability, neutralization value, octane value, etc., after sampling, immediately put into a brown bottle or the like, shield from light and it must be preserved in a dark place.
- (3) Space volume in sample container For provision against expansion or shrinkage, in the sample container a space volume of 5 % or more must be maintained.
- (4) Transportation of sample In order to prevent loss of liquid, vapor, etc. and mixing-in of water content and dust, to the glass bottle cover the plastic cap on the plug to seal tightly with sealing agent or cover the plug with a plastic sheet and bind rigidly at the portion of neck necessarily.