

Our requirements on the sampling and testing of Processed Fuel Oil supplied to the Quality Protocol standard in England and Wales

From 1st April 2011:

1. All PFO producers are required to have implemented a plan to sample to the IP475 method.
2. The site specific sampling plan must be approved in writing by the Environment Agency site inspector and will be assessed against the options in Appendix 1 below.
3. For in-house sampling, PFO producers must have their own ISO17025 UKAS accreditation and written confirmation from UKAS that an application has been made to add IP475 to their schedule.
4. External samplers can be used but they must have made an application to UKAS to add IP475 to their schedule. The external sampler must meet the requirements of the site specific sampling plan as approved by the Environment Agency. The contract between producer and external sampler should be sufficiently robust to ensure the sampling plan is met and that the relevant accreditation is pending.
5. All testing methods shall be carried out by a UKAS accredited laboratory to UKAS accredited methods. For IP592 (ICPMS metals apart from Hg) , IP593 (WDXRF metals apart from Hg), IP594 (Hg) and IP462 (PCBs) we may accept time-limited use of non-accredited methods. Such decisions will be taken on a site specific basis.

PFO Briefing Note 1
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APPENDIX 1 - Petroleum liquids - manual sampling. IP475-2005 (BS 2000-475)
Requirements of IP475 for sampling PFO (non volatile liquid from tanks at ambient pressure and below 200°C)
Check list for assessment of PFO sampling plan & procedures.

IP475 Section	Page no.	Requirement	Notes
5.2	8	Tank samplers	
	8	chains or synthetic fibre cords not recommended for suspending samplers	
5.2.2.1	8	Spot and zone samplers shall be constructed so that a sample can be taken at any specific level in a tank	Sampling device to be agreed in sampling plan
5.2.2.1	8	Spot sampling devices other than those detailed in IP475 are available and may be used	Sampling device to be agreed in sampling plan
5.2.5	20	Running samplers not acceptable for PFO	Not acceptable for PFO
5.2.6	20	All-level samplers not acceptable for PFO	Not acceptable for PFO
5.3	22	Drum and can samplers - not acceptable for PFO	Not acceptable for PFO
5.4	24	Pipeline samplers	
5.4.2	24	Positioning of sample probe within pipeline	Pipeline spot sampling not identified for tanks (7.1). Deviation to IP475 can be agreed in sampling plan only if producer can demonstrate the sample gives comparable results to a composite sample from top middle and lower samples.
5.4.2	24	Outlet valve delivery tube requirements to be agreed	
5.5	25	Sample receivers, vessels and containers	
5.5	25	Rinse reusable sample receivers in the fluid that is to be sampled to avoid contamination from previous samples.	To be assessed if appropriate and if not done justification included in sampling plan
5.5	25	Wherever possible, the sample should be transported to the laboratory in the vessel it was originally obtained in (the primary sample receiver), and therefore the methods which do not require sample transfer to a secondary vessel should be preferred	
5.5	25	Sample containers to be of a suitable material so integrity of sample not compromised	To be demonstrated by operator and container type stated in sampling plan
5.6	27	Container closures to be agreed	Closure type to be stated in sampling plan
5.7	27	Sample coolers - Not applicable for PFO	Not applicable for PFO
7	27	Procedures for homogenous petroleum liquids	Applicable to PFO
7.1	27	Unless otherwise specified, multiple spot samples shall be collected using the detailed procedure relevant to the particular application. Typically they are upper, middle and lower; or upper, middle and suction (outlet) samples	If sampling from pump circulation pipeline deviation to IP475 can be agreed in sampling plan only if producer can demonstrate the sample gives comparable results to a composite sample from top middle and lower samples.

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7.1	28	The minimum number of samples shall be in accordance with Table 1	To be specified in sampling plan
7.2	28	Precautions	
7.2.1.1	28	Avoid contamination of sample e.g. by rainwater or perspiration. Avoid sample transfer between containers	
7.2.1.2	28	Sampling personnel shall be fully instructed in the relevant procedures for the particular sampling application. Sampling procedures to be closely followed	
7.2.1.3	28	Samples shall not be drawn from unperforated or unslotted still wells, guide poles or standpipes.	
7.2.1.4	28	For handling samples, use equipment, containers, receivers or samplers that are impervious to and resistant to solvent action by the product handled (see 5.1)	
7.2.1.5	28	Thoroughly inspect all sampling equipment, including closures, to ensure that it is clean and dry	To be specified in sampling plan
7.2.1.6	28	Leave a minimum of 5% ullage in the container to allow for expansion. If spot samples are drawn from a tank, some of the sample has to be removed from the sample container, this shall be done immediately after the sample container has been withdrawn from the tank	To be specified in sampling plan
7.2.1.7	29	Immediately after filling and closing the sampler, sample receiver or container, examine it closely for leaks	To be specified in sampling plan
7.2.2	29	If samples are taken for the determination of trace elements e.g. lead, specially prepared sample containers may be required. Take such samples directly in the prepared container	To be specified in sampling plan
7.2.3	29	Volatile products - Not applicable for PFO	Not applicable for PFO
7.2.4	30	Tank side and pipeline sampling	
7.2.4	30	If tank side or pipeline sampling is used, adopt the following sampling precautions: a) completely flush sampling line to remove previous contents, b) sample outlet shall extend to near bottom of sample container. C) and d) not applicable to PFO	To be specified in sampling plan
7.2.5	30	Labelling and transport	
7.2.5.1	30	Clearly label containers: tie on labels preferred. Use indelible marking on labels. Information as specified in 7.2.5.1 to be marked on label	To be specified in sampling plan
7.3	30	Tank sampling	
7.3.1		Shore tanks	
7.3.1.1		Vertical cylindrical tanks	

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7.3.1.1.1	30	Spot Samples	
		Lower the sampling device until its opening is at the required depth, open it in the appropriate manner and maintain it at the required level until it is filled. Retrieve it and either decant a small portion back to the tank to create an ullage space before sealing the sample, or carefully transfer the complete sample to a secondary sample receiver.	To be specified in sampling plan
		Temperature conditioning of sampler not required for PFO	Temperature conditioning of sampler not required for PFO
		When sampling at different levels take samples in sequence from top to bottom, in order to avoid disturbance at low level.	To be specified in sampling plan
		In the case of a zone sampler (with essentially full bore top and bottom valves that allow the tank contents to flush through the sampler as it is lowered), lower the sampler in a controlled manner until it is at the required depth. Close the valve(s) as soon as the lowering operation ceases, and retrieve the sample immediately. Carefully transfer the complete sample to a secondary sample receiver.	To be specified in sampling plan
		Where the design of the zone sampler does not permit full flushing during lowering, it is recommended that the sampler be raised and lowered two or three times after reaching the spot sampling location, before closing the valve(s). The raising and lowering shall be through a distance of at least the height of the sampler.	To be specified in sampling plan
		In the case of a top sample, lower the open sampler/container carefully until its neck is just above the surface of the liquid, and then allow the sampler to fall sharply 150 mm below the surface. When the sampler is full, as indicated by the cessation of air bubbles, withdraw it and proceed as for regular spot samples.	To be specified in sampling plan
7.3.1.1.2	31	Composite samples	
		A composite sample may be prepared from representative sub samples of spot samples obtained from within a single tank (e.g. by combining sub samples from the upper, middle and lower spot samples). Composite samples shall include all the material collected in the primary sampling device without subdivision. The volume collected in the primary sampling device shall be chosen to allow the entire contents of the device to be added to the volume of the other sub samples in the transport container. Compositing of sub samples smaller than the entire contents of a sub sample shall only be performed in a laboratory with facilities to assure adequate mixing and measurement of the sub samples.	

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		To prepare either type of composite sample, transfer sub samples of representative individual samples into a composite-sample container, and mix them together gently. The sub samples shall be volume weighted in proportion to the quantity that each represents.	
		When the sub samples that are to be combined originate from a tank of non-uniform cross-sectional area (or from multiple tanks), the compositing operation shall require careful calculation and measurement of the sub samples to maintain the representative nature of the sample. These operations shall, if practical, be conducted under controlled laboratory conditions.	To be specified in sampling plan
		Do not prepare composite samples for testing unless they are specifically requested, and agreed by the interested parties. As an alternative to physical compositing, individual spot samples shall be tested and a mean value calculated from the individual test results in proportion to the bulk represented by each sample.	
7.3.1.1.3	31	Bottom sampling - Not required for PFO	Not required for PFO
7.3.1.1.4	32	Interface sampling - Not required for PFO	Not required for PFO
7.3.1.1.5	32	Tap (tank side) sampling - not acceptable for PFO	Not acceptable for PFO
7.3.1.1.6	32	All-level sampling - not acceptable for PFO	Not acceptable for PFO
7.3.1.1.7	33	Running samples - not acceptable for PFO	Not acceptable for PFO
7.3.1.2	33	Horizontal tanks with circular or elliptical cross-sections	
		Except when noted otherwise, samples shall be taken as spot samples as described in 7.3.1.1.1 from the levels indicated in Table 2. If they are to be combined to give a composite sample as described in 7.3.1.1.2, combine them in the proportions given in Table 2.	To be specified in sampling plan if applicable
7.3.1.3	32	Tanks with other geometrical shapes	
		Sample spherical tanks and tanks of irregular shape by taking spot samples as described in 7.3.1.1.1. Determine the actual levels at which the samples are to be taken to allow for the volume distribution over the height of the tank.	To be specified in sampling plan if applicable
7.3.1.4	34	Tanks fitted with vapour lock valves - not applicable to PFO	Not applicable to PFO
7.3.1.5	34	Pressurized tanks with valved sample points - not applicable to PFO	Not applicable to PFO
7.3.2	35	Tanks on ships or barges - not applicable to PFO	Not applicable to PFO currently
7.3.3	36	Railcars - not applicable to PFO	Not applicable to PFO currently
7.3.4	36	Road vehicle tankers	

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		For road vehicle tanks, if open sampling is acceptable, use the procedures described for sampling horizontal cylindrical or other geometry tanks as appropriate (see 7.3.1.2 and 7.3.1.3). If restricted or closed system sampling is required, use one of the procedures described for sampling tanks fitted with vapour-lock valves (see 7.3.1.4).	
7.4	36	Pipeline sampling	
		It is often necessary to obtain manual dynamic pipeline samples, e.g. for instrumentation verification and quality control purposes. It should be noted that such samples are spot samples which may or may not be representative of the bulk transferred quantity.	
7.4.2	36	Homogenous liquids	
		Sampling of homogeneous liquids shall be performed using a suitable pipeline sampling apparatus (see 5.4.2). Before a sample is drawn, flush the sample line and valve connection with the product to be sampled, after which draw off a sample into a sample container or receiver, taking into account the precautions given in 5.4, 7.2.4 and 7.4.3 as appropriate.	
7.4.3	36	Spot sampling of high vapour pressure liquids - not applicable for PFO	Not applicable for PFO
7.5	40	Dispenser (nozzle) sampling - not applicable for PFO	Not applicable for PFO
8	41	Procedures for crude oils and other non-homogenous petroleum liquids (additional procedures applicable to PFO)	additional procedures applicable to PFO
8.2.1	41	Tank sampling	
		Where it is necessary to assess the level of stratification within a tank's contents, draw samples initially from the upper, middle and lower (or suction) levels, transport them to the laboratory or testing location and test them individually for density, water and sediment content.	
		If the range of the results of these tests lie within ± 1 kg/m ³ (density) and $\pm 0,1$ % (V/V) (water content), the tank contents shall be considered as representative of the bulk, and the average results shall be taken.	
		If the range of the results of these tests does not lie within the specified limits, the tank contents are probably stratified. In these circumstances, additional spot samples shall, if possible, be taken at intervening or equidistant levels, and all the individual test results averaged. For this purpose, the samples for density, water, and sediment analysis shall not be physically composited, but the results of analyses on the separate samples may be mathematically composited.	For PFO protocol procedures can require additional mixing (processing) of the product until the density and water content requirements are achieved. Then taking of additional top middle and lower samples to make up a composite sample for full PFO analysis.

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9	43	Sample handling	
9.1	43	General	
9.1.2	43	The method of handling a sample will depend on the purpose for which it has been taken. The laboratory analytical procedure to be used will often require a special handling procedure to be associated with it. For this reason, consult the appropriate method of test so that any necessary instructions as to sample handling can be given to the person drawing the sample. If the analytical procedures to be applied have conflicting requirements, draw separate samples and apply the appropriate procedure to each sample.	
9.2	43	Homogenization of samples	
9.2.1	43	Introduction	
		Procedures are specified for the homogenization of samples that may contain water and sediment, or are in any other way non-uniform. Homogenization is required before any transfer of a partial quantity of sample, such as sub sampling or removal of a test portion. Care is required to ensure that the homogenization process does not in itself cause a loss of representativity, such as through the loss of light components. Procedures for verifying that the sample is satisfactorily mixed before transfer are given in 9.3.	Homogenization would usually be done at the third party laboratory if samples are not analysed in house
		It is not possible to manually agitate samples of liquids containing water and sediment sufficiently to disperse the water and sediment within the sample. Vigorous mechanical or hydraulic mixing is necessary in order to homogenize the sample prior to transfer or sub sampling.	
		Homogenization may be by various methods and will be governed by the sample, the sample receiver and/or the test method(s) used. Whichever method is used, it is recommended that the homogenizing system produces water droplets of sufficiently small size to ensure homogeneity and stability during subsequent handling, sub sampling and analysis procedures.	
9.2.2	43	Homogenization by high shear mechanical mixer	To be specified in sample plan. Evaluate proposed method against specified criteria and record on spreadsheet
9.3	45	Verification of (sample) mixing efficiency	
		Whatever means are chosen for obtaining a sub sample from a non-homogeneous mixture, verify the suitability of the mixing technique and the time required to obtain a homogeneous sample that can be representatively sub sampled.	To be specified in sample plan. Evaluate proposed method against specified criteria and record on spreadsheet

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9.4	47	Transfer of samples	
9.4.1	47	Transfer of sample between receivers/containers shall, if possible, be avoided unless conducted under controlled conditions. If the primary sample cannot be transported directly to the laboratory, it shall be transferred completely to a secondary container that can be transported directly to a laboratory. A complete transfer may be difficult due to evaporative loss of light components and/or the incomplete transfer of heavier components. In such circumstances, a sampling method shall be used that will permit the primary sample to be delivered to the laboratory without any transfer or sub sampling. If this is not possible, any sample handling and/or transfer outside of controlled laboratory conditions shall be kept to the absolute minimum.	
9.4.2	47	At every stage involving partial transfer of a sample, it is essential to homogenize the contents of the container from which the sample is being taken using one of the methods specified in 9.2.	
9.4.3	47	Verify the mixing efficiency for each combination of container and mixer by one of the methods specified in 9.3.	
9.4.4	47	Complete any transfer of sample within the period during which the mixture is known to be homogeneous. In some cases (especially during sub sampling into more than one container), it may be necessary to continue mixing during the transfer operations.	
10	47	Sampling of tank residues/deposits - Not required for PFO	Not required for PFO
11	47	Package sampling - Not applicable to PFO	Not applicable to PFO