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Petroleum and related products — Requirements and guidance for the maintenance of triaryl phosphate ester turbine control fluids

Pétrole et produits connexes — Exigences et recommandations pour la maintenance des fluides de régulation de turbines à base d'esters phosphates de triaryle

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*, Sub-committee SC 4, *Classifications and specifications*.

This first edition of ISO 11365 cancels and replaces ISO/TS 11365:2011.

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Introduction

Many turbine manufacturers or electrical power utilities have standardized requirements for the maintenance of triaryl phosphate ester hydraulic control fluids in service. A comparison of these requirements has made it possible to develop this document. However, the recommendations given in this document are for guidance and should not be considered as absolute. When interpreting results and deciding action, account has to be taken of various factors such as the conditions of use, the type of equipment, and the general trend in fluid characteristics.

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Petroleum and related products — Requirements and guidance for the maintenance of triaryl phosphate ester turbine control fluids

WARNING — The use of this document may involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this document to establish appropriate safety and health practice and determine the applicability of regulatory limitations prior to use.

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1 Scope

This document is applicable to the use of triaryl phosphate esters as fire-resistant fluids for turbine control and other hydraulic systems in power generation. These fluids fall under category HFDR of ISO 6743-4[1].

This document is intended to:

- assist the power equipment operator to maintain the fluid in a condition that will ensure the safe and reliable operation of the turbine while maximizing fluid life;
- recommend procedures for examining consignments of new fluid and monitoring the fluid in use;
- provide information on the safe handling, storage and disposal of the fluid;
- offer background information on the causes of fluid degradation.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3722, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods*

ISO 4021, *Hydraulic fluid power — Particulate contamination analysis — Extraction of fluid samples from lines of an operating system*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at http://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

4 Description of triaryl phosphate ester fluids

Triaryl phosphates are complex mixtures of products produced from the reaction of phosphorus oxychloride with substituted phenols, for example xylenol or tertiarybutylphenol, and have been used as fire-resistant fluids in power generation applications for over 55 years. While the early fluids were neurotoxic as a result of the presence of tri-o-cresyl phosphate, the products in commercial use for about the past 35 years have been free from this component and have very low levels of neurotoxicity. Modern phosphate esters are manufactured to meet the stringent health and safety requirements of CEN/TR 14489[2]. However, such requirements are subject to change and the fluid manufacturers should be contacted for the latest information. As with all chemicals, triaryl phosphates should be handled responsibly. The health and safety recommendations given in this document are intended to minimize exposure and to provide a margin of safety for workers handling these fluids.

Triaryl phosphate ester fluids normally have good resistance to oxidation, moderate to good air release and low foaming properties. However, they are sensitive to hydrolysis. These properties will, of course, slowly deteriorate during the life of the fluid. While some degradation can be tolerated without adverse effects on system performance, good monitoring procedures are necessary to determine when the properties have changed sufficiently to require action.

ISO 10050[3] is the standard for new triaryl phosphate ester hydraulic fluids when used in power generation applications.

For further information on the composition and technical performance of these fluids, the relevant technical literature available from the fluid manufacturers or suppliers should be consulted.

5 Health and safety precautions

5.1 Fluid handling

Triaryl phosphate ester fluids have extremely low vapour pressures and excellent stability, and under normal operating conditions give off no harmful vapours. There have been no reports of toxic effects through continued exposure, where sensible handling precautions have been taken. However, when handling phosphate esters all personnel should be aware of the nature of the material they are handling and consider the recommendations set out below.

— Accidental swallowing of the fluid or inhalation of vapours at elevated temperatures, which are the main potential sources of entry into the body, can be harmful and should be avoided. In event of ingestion, medical attention should be sought immediately.

— Goggles should be worn at all times when the fluid is being handled or when working on hydraulic control or other systems containing the fluid. Eye splashes may cause severe irritation. If any fluid gets into the eyes, they should be irrigated with water as soon as possible and medical attention sought.

— While the fluid is being handled, eating, drinking and smoking should be prohibited to prevent contamination of the lips and mouth. After handling the fluid, and before eating, drinking or smoking, the hands shall be thoroughly washed.

— Exposure to the skin normally represents minimal hazard and standard sanitary practices will prevent any adverse health effects. No ill effects have been reported from short-term skin exposure. However, continuous exposure to degraded fluid, which is acidic, should be avoided. This can be achieved using suitable impermeable protective gloves or barrier creams. Particular attention should be paid to the thorough cleaning of the skin and removal of any soiled clothing if extensive and prolonged contact with the fluid occurs.

— If the fluid leaks on to hot pipework, white fumes may be emitted. If these are inhaled, they can cause irritation of the throat and lungs. Therefore, when working in an environment where fumes are being emitted, breathing apparatus should be worn.

If required, further handling and safety information can be obtained from the fluid manufacturers or suppliers.

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5.2 Waste disposal

As far as is practicable, spillages shall be prevented from entering surface drainage channels. Fluid that has leaked from containers or from the system should be adsorbed onto sand, sawdust or other suitable adsorbent and disposed of in accordance with local regulations.

The disposal of used fluid in bulk should be carefully controlled to avoid contamination of the environment. All waste fluid shall be considered potentially harmful and be disposed of in accordance with local regulations.

6 Fire hazards

6.1 General points

Triaryl phosphate ester fluids do not readily ignite and do not support combustion but cannot be considered non-inflammable.

If the fluid leaks from the hydraulic system into lagging, it may decompose in the lagging with the emission of smoke and possibly result in a smouldering fire. Good ventilation of areas where fire hazards exist should therefore be provided.

The best method of avoiding a fire hazard is to prevent fluid leaks by following the operating and maintenance instructions, and by keeping the related equipment in a good state of repair at all times. In areas where experience has shown that leaks may develop, the following procedures will give additional protection:

— seal all the lagging material exposed to leaking fluid with finishing cement to provide a non-porous surface;

— cover exposed lagging with aluminium sleeves to prevent entry of fluid;

— provide drip trays to direct spilled fluid away from the lagging to collection points;

— replace any lagging material where the fluid has been absorbed into the insulation.

6.2 Methods of extinguishing fires

If a triaryl phosphate ester fluid is ignited as described above, the fire can be extinguished with foam, dry powder, carbon dioxide or water. However, if water is used, care should be taken to minimize direct contact with hot steel components, since it can cause rapid cooling with severe distortion or cracking. If used near electrical components, it may cause short-circuits and corrosion.

In the event of a lagging fire any damaged lagging should be removed, preferably when the pipe is cool and the fire extinguished. The lagging should then be replaced. If the lagging is cut away, it should be dropped into a container and covered to stop further smouldering, decomposition and the emission of smoke. Gloves, protective clothing and breathing apparatus should be worn when handling smouldering lagging.

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7 Compatibility of materials

7.1 Seals, paints and gaskets

Most seal materials, paints, and gaskets commonly found in hydraulic systems using petroleum-based fluids are not compatible with triaryl phosphate ester fluids. The use of unsuitable seals and gaskets can result in swollen or eroded materials, which may lead to fluid leaks or the binding of moving parts. Paints should be resistant to triaryl phosphate ester or the surfaces left unpainted. Some metals, such as copper and zinc, may promote fluid degradation, and their use should be minimized.

Materials currently recommended for use as seals are polytetrafluoroethylene (PTFE), fluorocarbon rubber (FKM) and ethylene propylene diene rubber (EPDM); hoses are normally made of butyl rubber (IIR). Suitable paints include stoved epoxy resins and vinyl ester-based products.

Attention is drawn to the fact that some materials, even those considered physically compatible, can adversely affect fluid performance. For example, silicone-based seals and gaskets should be avoided as they have an adverse effect on fluid foaming and air release properties.

If there is any doubt concerning the compatibility of replacement seals or hoses for the hydraulic system, the supplier of the fluid or the manufacturer of the system shall be consulted.

7.2 Electrical cable insulation

Triaryl phosphate ester fluid will soften and degrade some insulating materials, for example, polyvinyl chloride (PVC). Insulation containing PVC should therefore not be used in the vicinity of the control system. The fluid manufacturers recommend polytetrafluoroethylene, polyamide, polyethylene or polypropylene, but suppliers of the cable should be contacted in specific cases as many insulating materials and coatings are in use.

Care should be taken to avoid fluid spills onto cable insulation and in areas where spills may occur, the cabling should be shielded.

If cable insulation of unknown composition has accidentally come into contact with the fluid, it should be wiped clean with rags, preferably wetted in a solvent that will remove the fluid and not harm the insulation. Chlorinated solvents should not be used. Cables should then be inspected periodically to determine if they are suffering any deterioration.

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8 Factors affecting service life

8.1 General

Factors that affect the service life of triaryl phosphate ester fluids include:

- system design;
- system operating conditions;
- system maintenance;
- fluid contamination;
- fluid condition monitoring;
- fluid treatment or purification;
- fluid top up rate.

A more detailed explanation of these factors is provided below.

8.2 System design

There are a number of different control system designs in use. Several design factors impact upon the severity of operation of the control system, hence on fluid degradation. These include the following.

Pump type – constant volume pumps are the source of two problems:

a) Large volumes of fluid are continuously circulated at high flow rates over pressure relief valves. The fluid is heated in the valve and turbulence is often created as the fluid returns to the tank. This aerates the fluid and, at the elevated temperature, encourages degradation.

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b) If the fluid contains air bubbles these may be compressed in the pump with the generation of very high temperatures on the bubble walls. This process is known as dieseling.

Tank design – the discharges from warming and fluid return lines should be located below the lowest level of fluid in the tank and the return line should be located as far away from the pump inlet as possible. The design should ensure that any entrained air has adequate time to be released. This can be assisted by the appropriate use of sieves and baffles that direct fluid flow around the tank, and avoid a rapid move from the return line entry to the pump inlet.

Operating pressures – high pressures promote the rapid collapse of air bubbles in the pump and the development of very high temperatures at the bubble wall (dieseling). They will also result in higher temperatures as the fluid passes through the relief valve.

8.3 System operating conditions

Continuous turbine operation subjects the fluid to less stress than frequent stops and starts, while the use of tank heating (where fitted) or the operation of both circulating pumps concurrently can result in significant fluid degradation.

8.4 System maintenance

The quality of the fluid is adversely affected by, for example, any water leaks, the use of exhausted filters or incorrectly set relief valves.

8.5 Fluid contamination

8.5.1 General

As with most hydraulic fluids, triaryl phosphates are susceptible to contamination. The most common forms of contamination are indicated below in 8.5.2 to 8.5.6.

8.5.2 Air

Air, in either dissolved or dispersed form, is a common contaminant that causes the oxidative degradation of the fluid. Dissolved air can lead to the formation of varnish precursors and eventually to varnish itself if the precursors precipitate. Dispersed air (as bubbles) can cause dieseling when the bubbles are compressed in the pump with the generation of very high temperatures. Both mechanisms (but particularly dieseling) can result in the formation of significant amounts of small, even sub-micron, particulate together with acidic degradation products.

8.5.3 Water

Triaryl phosphate esters are susceptible to hydrolysis and the acidic degradation products formed further catalyse this reaction. In addition, the acidity developed may cause system corrosion, initiate servo-valve erosion and react with some adsorbent solid filters to produce gelatinous deposits in the system. Control of the water content is, therefore, particularly important. Desiccant breathers and, in some cases vacuum dehydrators or membrane dryers, are installed to minimize water contamination.

8.5.4 Particulates

Hydraulic control systems are sensitive to particulate contamination since very fine tolerances are found in some of the components. Where the fluid moves at high velocity, particulates can be abrasive and deposition in critical areas can impede system operation. They can be present in the system following assembly, in the original fluid or produced in service by wear, fluid degradation and/or system corrosion.

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In order to reduce particulate levels the system should be thoroughly flushed before use and new fluid added to the system should be pre-filtered through a 5 µm, ($\beta = 1\,000$) filter. In service, fine filtration is essential to maintain an acceptable level of the fluid cleanliness.

8.5.5 Mineral oil

Every effort should be made to avoid mineral oil contamination as this can adversely affect the fire resistance. Deposits may also form as a result of reaction between the triaryl phosphate ester and some mineral oil additives and these can lead to seizure of sensitive control elements. Mineral oil in small amounts can also adversely affect the fluid foaming and air release characteristics. Unlike other fluid contaminants that can normally be removed or reduced by *in situ* purification, mineral oil dissolved in the phosphate ester is impossible to eliminate by such methods.

8.5.6 Metal soaps

The use of some adsorbent filters to remove acid can introduce metal soaps into the fluid as a result of a chemical reaction between the acidic degradation products and components in the adsorbent medium. For example, calcium and magnesium carbonates in fuller's earth, and sodium and aluminium salts in activated alumina can result in the formation of metal phosphates. While low molecular weight soaps are usually soluble, their complexity and molecular weight can increase and, at some stage, they will precipitate. This tendency is greatest in cooler parts of the system, and where there is little or no flow.

Metal soaps often precipitate as gelatinous materials and blind filters/strainers, etc. Their presence will also increase the foaming tendency and air release value of the fluid. As a result, the use of these media is declining and ion exchange resins are often preferred.

8.5.7 Chlorinated materials

'Chlorine' contamination is normally due either to the use of a chlorinated cleaning solvent or to the presence of chloride in the atmosphere if the turbine is located close to the sea.

The presence of chlorine-containing products in the fluid can cause servo-valve erosion even at relatively low levels. The use of chlorinated solvents for system cleaning and of seawater for cooling the control fluid, should therefore be avoided. It should be noted that 'chlorine' is not a problem until it is converted to 'chloride' ion by fluid degradation. The total chlorine content in the fluid therefore represents the potential for developing erosion while the level of chloride ion actually present will directly affect the erosion process.

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8.6 Fluid condition monitoring

As a consequence of the possible contamination of the fluid and its degradation in use, regular monitoring of its properties is essential. Recommendations are usually available from the turbine builders, but in their absence, guidance is given later in this document.

8.7 Fluid purification

Unless action is taken to control the generation of acidity arising from hydrolytic or oxidative degradation, the system performance can be adversely affected and the fluid life significantly reduced. It is therefore essential to keep the fluid clean, with a low water content and acidity. Acceptable cleanliness is normally achieved by conventional in-line filtration and low water levels by vacuum dehydration or membrane drying. Acidity is controlled by adsorption filtration on a bypass loop to the main reservoir in which the fluid is continuously passed through an adsorbent medium (e.g. fuller's earth, activated alumina or ion exchange resin). A 2,5 µm, ($\beta = 1\,000$) fine particle filter is required downstream of the adsorption filter to ensure that no adsorbent particles are circulated. Fuller's earth and activated alumina are normally dried before use to increase their acid removal efficiency. Ion exchange resins may be used wet, or partially dried. Both forms of resin can release water into the fluid and any installed drying equipment should be able to maintain the water level within the recommended limits.

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Adsorbent solids can also remove other ionic material, for example chloride ions, and the filter unit as a whole assists in maintaining fluid cleanliness. With fuller's earth and activated alumina, it may not be possible to control the acidity if it is allowed to exceed ~0,3 mg KOH/g. In such circumstances, replacement of the complete fluid charge may be necessary.

8.8 Fluid top up

Some fluid is lost from the system through leakage and when changing filters, etc. This should be replaced and the fluid in the reservoir maintained at or about the maximum operating level. If the leakage rate is high the amount of fresh fluid being added to the system will help to maintain fluid quality. This action, however, is not a substitute for the regular monitoring of the fluid condition.

9 Delivery and storage

Fluid should be supplied in sealed drums. Delivery in bulk or semi-bulk containers is not recommended because it is difficult to control the cleanliness of the containers and the transfer/unloading system.

Drums shall preferably be stored inside in a dry location relatively free of dust and dirt. If kept outdoors it is essential that they are stored on their sides under cover with the two bungs horizontal to minimize moisture ingress. Any drum opened for sampling fluid should be resealed immediately, stored indoors and used as soon as possible. The long-term storage of partially filled drums is not recommended, in view of the increased risk of contamination. The fluid could, however, be used for top-up providing it has been checked for acidity, water and cleanliness before use. Empty drums should be resealed and stored indoors for future use in case the fluid in service is drained off during a full or partial replacement.

To avoid possible contamination from mineral oils, triaryl phosphate ester fluids should be clearly labelled and stored separately.

When transferring fluid from containers into the system the fluid should be filtered to ensure that contamination is avoided; a suggested filter rating is 5 µm, ($\beta = 1\,000$). If a transfer pump is used this should preferably be a dedicated unit, and cleaned before use. All seals and hoses should be compatible with the phosphate ester and the drum top (especially around the bungs), shall be cleaned before attaching the transfer pump.

10 Flushing the system

Before commissioning a new system, after system repairs or if a highly degraded charge has been removed from the system, it may be necessary to flush the circuit. This is to remove residual contamination, which if left in place could slowly dissolve in, or be circulated with, the operating charge and adversely affect its performance.

For this operation it is normally recommended to use a fluid of the same chemical type as the operating charge but not necessarily manufactured to the same high specification. Fluid suppliers should be able to advise on the most suitable product for flushing the system.

When using a flushing charge it is expected that all but a small amount (≤~3 %), of the flush fluid can be removed from the system after circulation, and that this residue will have little, if any, effect on the properties of the in-service fluid. If a large amount of fluid is likely to remain in the system after draining, another option would be to circulate the operating charge while monitoring the level of contamination. Only when this has dropped to an acceptable level and other fluid properties are in specification can normal use commence.

A system flush can consist of adding the charge to the system and circulating with all the components in place. This will remove some residues but is unlikely to clean the system thoroughly. If significant deposits from the previous charge are anticipated, the tank should be roughly cleaned before adding the flushing charge. It may be necessary to remove or bypass sensitive system components and use flushing valves on the actuators. Where possible the fluid flow in the pipework should be turbulent in order to remove as much of the deposits as possible and this may require the flushing of the system

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section by section. Normally, the turbine builder will have written flushing procedures and these should be followed.

11 Fluid sampling

11.1 General

When taking fluid samples from a reservoir or a system in service, it is important that proper sampling techniques are followed, particularly when measuring fluid cleanliness. The procedure should provide samples for laboratory analysis that are representative of the fluid in the system. Experience has also shown the need for standardized sampling procedures for sampling from containers when checking deliveries of new fluid.

The use of personnel trained in the implementation of the relevant ISO standards (ISO 3170, ISO 3722 and ISO 4021) is highly recommended as carelessness can introduce contaminants that will cause erroneous results.

Guidelines for suitable sampling and sample handling techniques are given in 11.2 to 11.5.

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11.2 Containers

Samples should be taken in a “suitable” container.

- The container should be compatible with triaryl phosphate ester. Some plastics, for example PVC, are not compatible.
- The container should be appropriate for whatever handling is required. Containers with leaking tops and unprotected glass containers are not suitable for shipment.
- The container should be of sufficient size. An extensive chemical analysis cannot be carried out if an insufficient sample is submitted. Normally 1 L is sufficient unless a larger quantity is requested. A separate 200 ml sample should be taken for a particle count analysis.

For particle count analysis it is essential to take samples from the circuit in specially cleaned bottles using standardized procedures and preferably from sampling valves located at appropriate points in the circuit. This will ensure that no foreign material is introduced that would interfere and distort the result (cleaning of the sample containers is carried out in accordance with ISO 3722 and the sampling procedure in accordance with ISO 4021). Use of different sampling points may help to identify where particles are being generated and whether the filters are working efficiently.

11.3 Sampling in service

11.3.1 General

In order to obtain a representative sample the hydraulic power unit should be in operation under steady-state conditions (normal operating temperature and rated pressure) to ensure the thorough mixing of the fluid. The minimum circulation time needed to accomplish this is about 1 h.

11.3.2 Sampling points

Suitable sampling points should be provided on the system to enable determination of the behaviour of system components. Typically, these would be located:

- before and after each main filter bank;
- in the LP return to the fluid reservoir;
- downstream of the adsorbent solid filter.

11.3.3 Precautions to be taken when sampling from a line

When sampling from a line, the precautions listed below should be taken.

- Keep all sampling points and connectors clean, fit blanking covers when not in use and check the security of connections before using.
- Before opening, wash the sampling valve outlet with filtered solvent from a wash bottle and allow to air-dry.
- Carefully release between 500 ml and 1 000 ml of fluid to purge the valve and lines before taking the sample, collect the fluid in a container.
- Remove the cap and polyethylene film (where used) from the sample container and immediately fill the container from the flowing stream. The sampling flow rate should be set to yield the sample volume in 20 s to 30 s. Do not allow the sample container and valve to come into contact. Under no circumstances should the sampling valve be operated or any fluid lines be touched while the container is being filled.
- Remove the container from under the flowing fluid before closing the sampling valve; immediately replace the polyethylene film over the container mouth and tighten the cap.
- Dispose of the waste fluid.

11.3.4 Sampling from a tank or reservoir

If, after cooling and settling, free water is found in a sample taken by the method outlined above then, depending upon the fluid temperature, there may also be free water present in the system. The following procedure is used for investigating free water in the reservoir:

- thoroughly clean the area around the reservoir cover plate and remove the bolts;
- lift the cover and dip or skim a sample from the surface of the fluid using a clean bottle attached to a rod, or a commercial ‘grab sampler’;
- check the cover gasket for damage and replace if necessary, re-bolt the cover in place.

Any free water will be evident in the sample as a layer floating on the surface of the hydraulic fluid. However, soluble water may also precipitate as the sample cools.

If surface water is found in the reservoir, immediate action shall be taken to find the point of entry and measures taken to avoid further contamination.

If a representative sample of the bulk fluid is required for general analysis, the sampler should be immersed, if possible to approximately half the liquid depth taking care to avoid baffles, sieves and getting too close to the pump inlet. The rest of the procedure should then be followed as above.

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11.4 Sampling of fresh fluid

Both drum and semi-bulk/bulk consignments should be sampled in accordance with ISO 3170, which recommends the number of samples to be taken dependent upon the number of containers delivered. In cases where the product is suspected of being non-uniform or is supplied as a mixture of different batches, a larger number of drums or other containers should be sampled.

A representative sample would normally be taken from the middle of the container, but if contamination is suspected the fluid sample should be obtained from the point(s) most indicative of contamination by water or particulates, i.e. at the top and/or bottom of the drum or tanker compartment, providing the container has not recently been moved. All samples should be examined immediately for appearance.

Where post-delivery testing is undertaken, an additional sample shall be taken and reserved for future analysis by the fluid supplier or an alternative laboratory to that of the receiving station.

11.5 Labelling

Samples should be clearly labelled and include at least the following information:

a) customer name, if appropriate;

b) site and unit/turbine number;

c) circuit description/serial number;

d) fluid name;

e) fluid service hours;

f) fluid volume in service;

g) fluid make-up quantity since the last analysis;

h) sampling point;

i) date of sample;

j) type of purification system;

k) health and safety labels, where appropriate.

12 Recommended procedures for fluid monitoring

12.1 Appearance and colour

These tests involve a visual assessment of the clarity, freedom from air bubbles and significant particulate content in the fluid. Where colour is measured, the recommended test method is ISO 2049[4].

They are quick and simple tests to carry out but can tell the operator whether any significant change has occurred since the last sampling. For example, if the fluid shows signs of turbidity, this could be due to a high water content or the precipitation of degradation products. The checks can also be indicative of longer-term problems. If the colour is seen to be changing quite rapidly, then oxidation (possibly even dieseling) may be occurring.

Table 1 indicates recommended corrective action dependent upon the appearance of the used fluid immediately after sampling and after standing for an hour.

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Table 1 — Visual inspection of the samples

Appearance of the oil just after sampling	Appearance of the oil after 1 h	Probable cause	Action to be taken
Clear	Clear	-	-
Foam at the surface	Foam collapsed	Air entry in the circuit	Seek origin of air and control
	Persistent foam	Loss of antifoam or contamination of fluid	Control foaming
Sample cloudy and becoming clear from the bottom	Clear	Aeration	Seek origin of air and control
	Persistent cloudiness	High air entrainment	Seek origin of air and control
Sample cloudy	Clear, or slightly opaque, supernatant water layer	Unstable emulsion	Seek origin of water and control
	Milky	Stable emulsion	Seek origin of water and control
Dirty	Presence of decanted solid particles	Contamination, filtration problem	Seek origin of dirt and control
Rapid and unusual darkening	-	Contamination or excessive degradation (dieseling)	Seek origin of dirt and/or air and control

12.2 Water content

The recommended test method is ISO 760[5]. However, some additives present in oils may interfere with the Karl Fischer reagent, giving rise to erroneous results. In case of doubt or dispute, ISO 20764[6] should be used.

As well as the normal ingress of atmospheric moisture, water can enter hydraulic systems due to:

— seal failure;

— as a result of faulty topping-up procedures;

— ineffective reservoir breathers;

— through the use of 'wet' adsorbent media;

— cooler leaks.

If high levels of moisture are observed, and there is no evidence of abnormal ingress of water, then any dehydration equipment used may be inadequate or is malfunctioning.

Recommended corrective actions are as follows:

— ensure that there are no leaks past the desiccant breather;

— check the desiccant is not exhausted, replace if necessary;

— check the fluid coolers for leaks.

If leaks are discovered consult the manufacturer for repair procedures.

— check the reservoir covers are properly sealed;

— if a charge of fluid is badly contaminated with water, a vacuum dehydration unit is the quickest way to dry the fluid, but gross contamination may require replacement of the fluid charge or siphoning off the layer of free water in the tank;

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- where applicable, check that the tank exhauster is maintaining the desired vacuum above the fluid in the tank or that the membrane dryer is removing moisture from the air.

NOTE If ion exchange resins are used these will release water into the fluid immediately downstream of the resin. This is quite normal, but measures to remove the excess water (drying/dehydration) may need to be permanently installed.

12.3 Neutralization number (Acid number - AN)

This property is the most important test parameter used for monitoring the fluid condition, the recommended test method is ISO 6619[7].

An increase in acidity may be caused by degradation of the fluid by moisture, oxidation at high temperatures, chemical contamination of the system, or by the ineffective operation of any adsorption filters.

Recommended corrective actions are as follows:

- investigate and, if necessary, reduce the water level; correct any water leaks;
- change the adsorption filters and take fluid samples every 48 h until the neutralization number returns to an acceptable level.

For used fluids the potentiometric method is preferred because of the difficulty in obtaining a definite colour change in dark fluid. The colorimetric method can be used on fresh fluid, but for comparability of results it is recommended that the potentiometric method be used in both cases.

12.4 Fluid cleanliness

The methods used for particle counting are:

- ISO 4405 (gravimetric method)[8];
- ISO 4407 (microscope method)[9];
- ISO 11500 (automatic particle counting)[10];
- ASTM D7596 (direct imaging analysis)[11].

The calibration of the automatic particle counters is performed using ISO 11171[12] and the level of contamination is reported according to ISO 4406[13].

Most routine analyses involve the use of automatic counters. However, in cases of dispute, or where more information is required on the type of contaminant, particularly if significant amounts of particles ≤ 4 µm are thought to be present, consideration may be given to the use of the alternative procedures.

It is essential to take fluid samples from the circuit in specially cleaned bottles using standardized procedures and sampling valves in the circuit. This will ensure that no foreign material is introduced that would interfere and distort the result (cleaning of the sample containers is carried out in accordance with ISO 3722 and the sampling procedure in accordance with ISO 4021).

Any increase in particulate levels may be due to external contamination entering the system in added make-up fluid, or through seal failure. It can also be caused by inadequate maintenance, failure of a filter element, wear in a system component or degradation of the fluid.

Recommended corrective actions are as follows:

- ensure that any make-up fluid added to the system meets the manufacturer's specification for new fluid cleanliness;
- check the reservoir to ensure that all covers are in place and are properly sealed;

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- ensure filters have the recommended rating for the system;
- change filters and strainers if blocked;
- check carry-over of adsorbent solids;
- check for possible operating problems and wear of servo-valves, solenoid valves, pedestal trip valves, and pump or accumulator parts;
- consider additional filtration to restore the cleanliness of the fluid.

NOTE When carrying out maintenance on the system, it is important to observe strict cleanliness procedures.

12.5 Direct current resistivity (volume resistivity)

The recommended test method is IEC 60247[14].

A reduction in the d.c. resistivity of a fluid may be caused by an increase in acidity and/or moisture content, topping-up with an unsuitable grade of fluid, contamination with dirt or an electrically conducting material.

Recommended corrective actions are as follows:

- investigate and, if necessary, reduce the water level; correct any water leaks;
- use only recommended fluids as make-up;
- change adsorption filters;
- check fluid cleanliness.

12.6 Chlorine content

The recommended test method is microcoulometry (EN 14077[15]) but X-ray fluorescence (ISO 15597[16]) may also be used.

'Chlorine' originates mostly from the use of chlorinated cleaning solvents or from chloride ion in the atmosphere or cooling water. The effect of 'chlorine' contamination may be servo-valve erosion.

Recommended corrective actions are as follows:

- discontinue the use of chlorinated solvents around the hydraulic system;
- investigate coolers for leaks;
- change adsorption filters and take fluid samples every 48 h until the 'chlorine' content is reduced to within the turbine manufacturer's recommendations.

12.7 Mineral oil content

Several test methods may be used. For example, density, specific gravity (relative density), refractive index, thin layer chromatography and high-pressure liquid chromatography. However, if the amount of oil present is required the chromatographic methods may be preferred, as the others require a knowledge of the specific gravity or refractive index of the contaminating product. Density or specific gravity measurements will also not detect the presence of small amounts of mineral oil, typically less than 2 %. If in doubt, consult the fluid supplier.

The density of mineral oil is less than that of triaryl phosphate ester, and if present in excess of the solubility level, will float on top. However, unless very high levels are present, mineral oil will usually dissolve in the phosphate at operating temperatures but then (depending upon the amount present and the type of oil used) precipitate at ambient.

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Mineral oil contamination is usually due to topping-up with the wrong fluid, residual contamination from component tests with mineral oil, or the failure of common seals between the hydraulic system and plant items using separate mineral oil lubrication.

The main risk with mineral oil contamination is a reduction in the fire resistance of the phosphate ester. There can also be damage to any hydraulic accumulator bladders made of butyl rubber, as these are not compatible with mineral oil. Lastly, the presence of even small amounts of mineral oil can have an adverse effect on fluid air release properties.

Unfortunately, there is no easy way to remove mineral oil from phosphate ester and if oil is thought to be adversely affecting the fluid properties, it may be necessary to replace the complete fluid charge. To avoid this, only recommended fluids should be used, as make-up and fluid in any partly filled drum should be checked before adding to the system.

If mineral oil contamination is suspected, contact the fluid manufacturer or supplier for specific advice.

12.8 Viscosity

The recommended test method is ISO 3104[17].

A significant change in viscosity is normally due to fluid contamination. For example, water will cause a reduction in viscosity dependent upon the amount dissolved. In normal use it is most unlikely that phosphate viscosity will increase significantly as a result of oxidation.

Recommended corrective actions are as follows:

- check nature of contamination;
- if water, dry the fluid;
- if contaminated by oil or another (non-phosphate) synthetic fluid, consider a fluid change.

12.9 Air release

The recommended test method is ISO 9120[18].

The increase in air release value may be due to contamination (e.g. by dirt or mineral oil) or as a result of fluid degradation.

Recommended corrective actions are as follows:

- investigate and correct any contamination;
- check filters are operating effectively and, if exhausted, replace.

12.10 Foaming

The recommended test method is ISO 6247[19].

An increase in foaming may be due to the loss of antifoam or to the contamination of the fluid by degradation products, mineral oil or very fine particles. However, foaming is also very sensitive to system design and operation, and poor results in a static laboratory test are not necessarily indicative of a problem. If a high foaming tendency (>300 ml) or any stable foam is found in the laboratory test, it is necessary to check if significant foaming is occurring in the tank.

Recommended corrective actions (if foaming is found in the tank) are as follows:

- investigate and correct any contamination;
- check if the antifoam has depleted; if so, it may be necessary to at least partially replace the fluid charge as satisfactory antifoam dispersion *in situ* is very difficult to achieve.

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13 Examination of new fluid

All fluid is purchased against a manufacturing specification that normally meets the requirements of the different turbine builders, utilities and/or national and international standards organizations. However, there can be significant differences in their technical requirements and limits. The user should therefore be aware of the specification(s) relevant to the equipment involved.

Although deliveries of new triaryl phosphate ester fluid should always be accompanied by a certificate of analysis/conformity, a delivery should also be inspected to guard against the supply of an incorrect product. It is advisable to sample the consignment and to test it against the specification. ISO 3170 gives guidance on the number of samples required.

When fluid is supplied in drums, the tests should be performed on a composite sample particularly if the delivery is made up of different batches. Individual drum samples shall be retained until the analysis of the composite sample has been completed and found satisfactory.

If sampling suggests the material is outside specification then further investigation will be required. ISO 4259[20] gives guidance on how to judge the quality of a product with regard to specification limits and test method precision. When a drum is opened to remove a sample, this shall be carried out under clean and dry conditions according to ISO 3170 and the drum immediately resealed. Priority should be given to the use of any drum that has been opened, but if a long time elapses before use, the fluid condition should be checked before adding to the system.

If the material is supplied in bulk or semi-bulk containers, each tanker compartment or container should be sampled. If these are clear of debris and water, then the samples can be combined for a subsequent laboratory analysis of the consignment. As well as sampling individual tanker compartments, further sample(s) should be taken from the outlet of the flexible pipework, or at least from the tanker bottom valve manifold; the sampling flow rate should be set to yield the required sample volume in 20 s to 30 s.

When fluid is delivered in bulk, the acceptance tests may need to be performed before the tanker leaves the site. Whether the tanker remains until testing is complete or leaves immediately the product is discharged, is subject to negotiation between the supplier and user. However, consideration will need to be given to the risk of accepting a product out of specification and to the ease of recovering the charge before being put in service.

Normally the shelf life of fluid in sealed drums is up to two years and during that time it may very slowly deteriorate. While it is advantageous to check material before use if held in stock for over a year, it becomes essential if this period exceeds two years. If the product is found to be slightly outside specification (e.g. on acidity) its use may still be possible as adding a small quantity to a larger volume of in-specification fluid would be unlikely to have a significant adverse effect on bulk fluid properties and the fluid will still be subject to online conditioning. If in doubt, consult the fluid manufacturer or supplier.

13.1 Acceptance tests for new fluid

The minimum requirements for batch acceptance at the time of delivery are given in Table 2.

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Table 2 — Minimum acceptance requirements for a new delivery of triaryl phosphate ester fluid

Test methods	Properties	Test limits
Visual	Appearance	Bright and clear
ISO 3104	Viscosity	To comply with purchasing specification
ISO 6619	Acid number	
ISO 760	Water Content	
ISO 11500 and ISO 4406	Cleanliness[a]	

[a] The precision of cleanliness measurements by automatic counting methods is not stated. While repeatability may be acceptable, reproducibility can be affected by many factors and may lead to disputes. In case of disagreement, reference should be made to a direct imaging analysis method, for example, ASTM D7596[11].

13.2 Acceptance requirements after installation of a new fluid charge

A new fluid charge is filled into the system on commissioning a new turbine or when a fluid charge has to be replaced due to degradation/contamination.

In both cases the fluid will be transferred from drums/containers (or even a holding tank) to the system. Where transfer pumps are used, they should be clean, fitted with a 5 µm, (β = 1 000) fine particle filter and the seals/hoses should be compatible with the fluid being transferred. If significant contamination is expected within the system it may be necessary to flush it with the operating charge or a special flushing fluid. Further information on flushing is given in Clause 10.

After draining any flushing charge, the operating charge is filled into the system and circulated for approximately 24 h when a 2 l sample is taken for analysis. The extent of the testing will depend upon the requirements of the turbine builder or end user, but will normally include the methods mentioned in Table 2 with the possible addition of tests for mineral oil, chlorine content and resistivity. The results should be compared against the specification for new fluid.

Some slight deterioration in properties in comparison with the delivered fluid is to be expected if the system has not been flushed and there is a residue from the previous fluid charge. If doubt exists as to whether the fluid is in a satisfactory condition for continued use, consult the turbine manufacturer or fluid supplier.

14 Examination of used fluid

14.1 General

While essential for monitoring fluid performance, testing is time-consuming and can be expensive. The selected tests enable the extent of degradation/contamination to be identified quickly and ensure that its use can continue, at least until the next planned sampling, without a significant drop in performance or an adverse effect on the system. To reduce the cost of testing the critical tests are performed most frequently. Yet others can be used intermittently or when problems arise as there is normally little change in the property.

Tests therefore fall into the following two categories.

- Those that are performed on every sample and are usually undertaken by the power station laboratory. Typically these are the same as used for checking new fluid quality (see Table 2). The frequency of testing is usually at least monthly, although this can vary depending upon system design, operating conditions or if problems are being encountered.
- Those that are carried out less frequently, which are often more specialized procedures and may involve an external laboratory - perhaps that of the fluid supplier or manufacturer.

Where possible the tests used should have precision statements. The operator should be aware of the impact of precision on the validity of any conclusions drawn from the results.

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