DD ISO/TS 11365:2011

BSI Standards Publication

Petroleum and related products — Guidance for the maintenance and use of triaryl phosphate ester turbine-control fluids

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National foreword

This Draft for Development is the UK implementation of ISO/TS 11365:2011.

This publication is not to be regarded as a British Standard.

It is being issued in the Draft for Development series of publications and is of a provisional nature. It should be applied on this provisional basis, so that information and experience of its practical application can be obtained.

Comments arising from the use of this Draft for Development are requested so that UK experience can be reported to the international organization responsible for its conversion to an international standard. A review of this publication will be initiated not later than 3 years after its publication by the international organization so that a decision can be taken on its status. Notification of the start of the review period will be made in an announcement in the appropriate issue of *Update Standards.*

According to the replies received by the end of the review period, the responsible BSI Committee will decide whether to support the conversion into an international Standard, to extend the life of the Technical Specification or to withdraw it. Comments should be sent to the Secretary of the responsible BSI Technical Committee at British Standards House, 389 Chiswick High Road, London W4 4AL.

The UK participation in its preparation was entrusted to Technical Committee PTI/7, Lubricants and process fluids.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Petroleum and related products — Guidance for the maintenance and use of triaryl phosphate ester turbine-control fluids

Pétrole et produits connexes — Lignes directrices pour la maintenance et l'utilisation des fluides de régulation de turbines à base d'esters de triaryl phosphate

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TS 11365 was prepared by Technical Committee ISO/TC 28, *Petroleum products and lubricants*, Subcommittee SC 4, *Classifications and specifications*.

This first edition of ISO/TS 11365 is a revision of IEC 60978:1989, which was previously maintained by IEC/TC 10, *Fluids for electrotechnical applications*.

Introduction

Many turbine manufacturers and electrical power utilities specify limits on the properties of triaryl phosphate ester hydraulic control fluids in service. Some companies also provide recommendations on the action necessary if these limits are approached or exceeded, but few provide detailed guidance on fluid maintenance and use.

This Technical Specification identifies typical performance limits for used fluids and also contains detailed recommendations on their use and maintenance. While the requirements of the equipment builder and/or operator take precedence, particularly during any warranty period, the content of this Technical Specification can be read in conjunction with manufacturer/operator requirements, or provide guidance in their absence.

Petroleum and related products — Guidance for the maintenance and use of triaryl phosphate ester turbine-control fluids

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

1 Scope

This Technical Specification applies to the use of triaryl phosphate esters as fire-resistant fluids for turbine control and other hydraulic systems in power generation.

This Technical Specification is intended to

- help power equipment operators appreciate the important properties of triaryl phosphate esters;
- provide information on their safe handling, storage and use.

This Technical Specification addresses the causes of fluid deterioration and sets out procedures for examining consignments of new fluid, for monitoring the fluid in use and for maintaining an adequate fluid condition.

2 Normative references

The following referenced documents are indispensable for the application 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 3170:2004, *Petroleum liquids — Manual sampling*

ISO 3722:1976, *Hydraulic fluid power — Fluid sample containers — Qualifying and controlling cleaning methods*

ISO 4021:1992, *Hydraulic fluid power — Particulate contamination analysis — Extraction of fluid samples from lines of an operating system*

3 Description of triaryl phosphate ester fluids

Triaryl phosphates are complex mixtures of products produced from the reaction of phosphorus oxychloride with either xylenols or substituted phenols and they have been used as fire-resistant fluids in power generation applications for over 50 years. While the early fluids were neurotoxic as a result of the presence of tricresyl phosphate, the products in commercial use for about the last 30 years do not contain this component and have very low levels of neurotoxicity. Modern phosphate esters are manufactured to meet the stringent health and safety requirements of the 7th Luxembourg Report[24] and its more recent replacement, CEN/TR 14489. However, as with all chemicals, they should be handled responsibly. The health and safety recommendations given in this Technical Specification are therefore 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 can be sensitive to hydrolysis. These properties will slowly deteriorate during the life of the fluid. While some degradation can be tolerated without adverse effects on the functioning of the system, good monitoring procedures are necessary to determine when the properties have changed sufficiently to warrant action.

ISO 10050 is the technical specification 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 should be consulted.

4 Health and safety precautions

4.1 Fluid handling

Triaryl phosphate ester fluids have extremely low vapour pressures and excellent stability, and at normal operating temperatures 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 know the recommendations set out below.

- Accidental swallowing of the fluid or inhalation of vapours, 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 should be 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 washed thoroughly.

- Exposure to the skin normally constitutes a 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 highly degraded fluid, which is acidic, should be avoided. This can be achieved by using suitable impermeable protective gloves. 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 onto hot pipe work, white fumes may be emitted. If these are inhaled, they can cause irritation of the throat and lungs. When working in a fume-filled environment, breathing apparatus should therefore be worn.

4.2 Waste disposal

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

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

5 Fire hazards

5.1 General points

The 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 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 the customer's operating 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 soaked up by the fluid, because at high temperatures an exothermic reaction can occur leading to a temperature rise and possibly a smouldering fire.

5.2 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 - and 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.

6 Compatibility of materials

6.1 Seals, paints and gaskets

Most seal materials, paints, and gaskets commonly found in hydraulic systems using petroleum-based fluids are not compatible with the 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.

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. Some materials currently used for seals are polytetrafluoroethylene (PTFE), fluorocarbon rubber (FKM) and ethylene propylene diene rubber (EPDM) while butyl rubber (IIR) hoses are recommended. 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.

6.2 Electrical wire insulation

Triaryl phosphate ester fluid will soften and eventually decompose some insulating materials. For example, polyvinyl chloride (PVC) may contain a triaryl phosphate ester as a plasticizer; soaking PVC in this fluid will therefore soften it. 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 cable manufacturers should be contacted in specific cases as many insulating materials and coatings are in use.

The most effective way of preventing problems is by avoiding spills of fluid onto electrical wiring insulation. In areas where spills onto wiring may occur (e.g. during maintenance), the wiring should be shielded.

If wiring insulation of unknown composition has been accidentally wetted with the fluid, it should be wiped clean with rags, preferably wetted in a solvent that will remove the fluid (e.g. isopropanol) 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.

7 Factors affecting service life

7.1 General

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

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

7.2 System design

There are a number of different control system designs in use. Several design factors impact on the severity of operation of the control system and hence on fluid degradation. These include pump type, tank design and operating pressures, as described below.

- **Pump type** Constant volume pumps are the source of two problems, as outlined below.
    1) If the fluid contains air bubbles these may be compressed in the pump with the generation of very high temperatures on the bubble walls.
    2) 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 encourages rapid degradation.

- **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 return line to the pump inlet.

- **Operating pressures** High pressures promote the rapid collapse of air bubbles in the pump and 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.

7.3 System operating conditions

Continuous operation subjects the fluids to less stress than if the unit is subjected to frequent stops and starts.

7.4 System maintenance

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

7.5 Fluid contamination

7.5.1 General

As with most hydraulic fluids, triaryl phosphates are susceptible to contamination. The most common forms of contamination are outlined in 7.5.2 to 7.5.6.

7.5.2 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 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 are installed to minimize water contamination.

7.5.3 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.

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.

7.5.4 Mineral oil

Every effort should be made to avoid mineral oil contamination, as this may 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.

7.5.5 Metal soaps

The use of some adsorbent filters to remove acid can introduce metal soaps into the solution, 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 (present as sodium aluminates) in activated alumina can be released into the fluid as 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 collect 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, these media are used less frequently and ion exchange resins are preferred instead.

7.5.6 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 in the fluid of chlorine-containing products can cause servo-valve erosion even at relatively low levels. The use of chlorinated solvents for system cleaning and of sea-water 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. The total chlorine content in the fluid therefore represents the potential for developing erosion, while the level of chloride ion actually present will directly impact the erosion process.

7.6 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; if such recommendations are absent, however, reference can be made to guidance given later in this Technical Specification.

7.7 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. This is normally achieved by conventional filtration in-line and by adsorption filtration on a by-pass loop to the main reservoir, in which the fluid is continuously passed through an adsorbent medium (e.g. fuller's earth or activated alumina). A 2,5 µm (β = 1 000) fine particle filter is required downstream of the adsorption filter to ensure that no adsorbent solid particles are circulated.

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

In order to avoid the release of metal soaps, ion exchange resins are now widely used to control acidity. However, they do release water into the fluid, the level of which needs to be controlled by vacuum dehydration, etc.

7.8 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. However, this action is not a substitute for the regular monitoring of fluid condition.

8 Delivery and storage

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

The fluids shall be stored inside in a dry location relatively free of dust and dirt. Empty drums should be resealed and stored for future use, in case the fluid in service is drained off during a full or partial replacement. Any drum opened for sampling fluid should be resealed immediately and used as soon as possible.

To avoid confusion, triaryl phosphate ester fluids should be clearly labelled and kept separately from mineral oils.

When transferring fluid from containers into the system, the fluid should be filtered (a suggested filter rating is 5 µm, $\beta = 1\ 000$) to ensure that contamination is avoided. If a transfer pump is used, this should preferably be a dedicated unit. Alternatively, care should be taken to avoid contamination from the previous product and to ensure that all seals and hoses are compatible with the phosphate-ester.

9 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 by, the operating charge and adversely affect its performance.

For this operation, it is normally advisable 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.

The use of a flushing charge assumes that all but a small amount can be removed from the system after circulation, so a small remaining quantity (~3 %) 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 the 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, much as would be done with the operating charge. 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 to use flushing valves on the actuators and much coarser filters. Where possible, the fluid flow in the pipe work should be turbulent in order to remove as much of the deposit as possible, and this may require flushing of the system section by section. Normally, the turbine builder will have written flushing procedures and these should be followed.

10 Fluid sampling

10.1 General

When taking fluid samples from a reservoir or a system in service, it is important that proper sampling techniques be followed, particularly when measuring the particulate content. 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 used for the quality control of supplies of unused fluid.

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

Suggested guidelines for proper sampling and sample handling techniques are given in 10.2 to 10.5.

10.2 Containers

Samples should be taken in a suitable container, i.e. the container should be:

- **compatible** with triaryl phosphate ester (some plastics, e.g. PVC, are not compatible);
- **appropriate** for whatever handling is required (containers with leaking tops and unprotected glass containers are not suitable for shipment);
- **of sufficient size** (an extensive chemical analysis cannot be carried out if 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 special 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. See ISO 3722 for the cleaning of the sample containers and ISO 4021 for the sampling procedure. Use of different sampling points may help to identify where particles are being generated and whether the filters are working efficiently.

10.3 Sampling in service

10.3.1 General

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

10.3.2 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.

10.3.3 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; check the security of connections before using.
- Before opening, wash the sampling valve outlet with filtered solvent from a wash bottle.
- Allow between 500 ml and 1 000 ml of fluid to purge the valve and lines before taking the sample; catch the fluid in a container and discard.
- Remove the cap and polyethylene film 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 touch. 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 sampling valve; immediately replace the polyethylene film over the container mouth and tighten the cap.

10.3.4 Sampling from a tank or reservoir

If, after cooling and settling, free water is found in a sample taken by the method outlined above, there may also be free water present in the system, depending on the fluid temperature. The following procedure is used to investigate 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.

10.4 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 on the number of containers delivered. In cases where the product is suspected of being non-uniform, a larger number of drums should be sampled.

A representative sample would normally be taken from the middle of the container, but if contamination is suspected, the fluid sample would be obtained from the point(s) most indicative of contamination by particulates or water, i.e. at the top and the 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, if necessary, for future analysis by the fluid supplier or an alternative laboratory to that of the receiving station.

10.5 Labelling

Samples should be properly labelled. Marking should 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.

11 Recommended procedures for fluid monitoring

11.1 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 preferred test method is ISO 2049.

The tests are quick and simple to carry out, but they can inform the operator as to whether any significant change has occurred since the last sampling. For example, if the fluid shows signs of turbidity, then 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 on the appearance of the used fluid immediately after sampling and after standing for 1 h.

Table 1 — Visual inspection of the samples

Appearance of the oil		Probable cause	Action to be taken
Immediately after sampling	**After standing 1 h**		
Clear	Clear	—	—
Foam at the surface	Foam collapsed	Air entry in the circuit	Seek origin of air and control
	Persistent foam	High foaming tendency	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 water emulsion	Seek origin of water and control
	Milky	Stable emulsion with water	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 and control oxidation

11.2 Water content

The recommended test method is ISO 760. 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 should be used.

As well as the normal ingress of atmospheric moisture, water can get into hydraulic systems due to seal failure, as a result of faulty topping-up procedures, ineffective reservoir breathers, through the use of “wet” adsorbent media or a cooler leak. 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;
- where 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 the siphoning-off of the layer of free water in the tank;
- where applicable, check that the tank exhauster is maintaining the desired vacuum above the fluid in the tank.

NOTE Where 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 (dehydration) might need to be permanently installed.

11.3 Total acid number (TAN)

The recommended test method is ISO 6619.

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

Recommended corrective actions are as follows:

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

NOTE 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 and light coloured fluid, but for consistency, it is advisable that the potentiometric method be used in both cases.

11.4 Fluid cleanliness

The methods used for particle counting are:

- ISO 4405 (gravimetric method)
- ISO 4407 (microscope method)
- ISO 11500 (automatic particle counting).

The calibration of the automatic particle counters is performed using ISO 11171 and the level of contamination is reported in accordance with ISO 4406.

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, 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 special sampling points in the circuit. This will ensure that no foreign material is introduced that would interfere and distort the result. See ISO 3722 for the cleaning of the sample containers and ISO 4021 for the sampling procedure.

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 oxidation of the fluid.

Recommended corrective actions are as follows:

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

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

11.5 Direct current resistivity (Volume resistivity)

The recommended test method is IEC 60247.

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

Recommended corrective actions are as follows:

- investigate and correct any water leaks;
- use only recommended fluids as make-up;
- change adsorption filters;
- check fluid cleanliness.

11.6 Chlorine content

The recommended test method is microcoulometry (NF EN 14077), but X-ray fluorescence (ISO 15597) may also be used.

“Chlorine” originates mostly from the use of chlorinated cleaning solvents or from chloride ion in the atmosphere. The result 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.

11.7 Mineral oil content

Several methods may be used, e.g. specific gravity, refractive index, thin layer chromatography and high pressure liquid chromatography. However, if the amount of oil present is required, chromatographic methods may be preferred, as the others require a knowledge of the specific gravity or refractive index of the contaminating product. 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 on the amount present and the type of oil used) precipitate at ambient temperature.

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

The main risk with mineral oil contamination is that there may be a reduction in the fire resistance and possible damage to any hydraulic accumulator bladders which, if made of butyl rubber, are not compatible with mineral oil. Furthermore, 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 the 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.

Recommended corrective actions are as follows:

- contact the fluid manufacturer for specific advice.

11.8 Viscosity

The recommended test method is ISO 3104.

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

Possible corrective actions are as follows:

- check the nature of the contamination;

- if water, dry the fluid;

- if contaminated by oil or by a synthetic fluid, consider a fluid change.

11.9 Air release

The recommended test method is ISO 9120.

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 the filters are operating correctly; if exhausted, replace.

12 Examination of new fluid

12.1 General

All fluid is purchased against a manufacturing specification which normally meets the requirements of the different turbine builders, utilities and/or national and international standards organizations. However, not all standards are alike in requirements or limits. The user should therefore be aware of the specification(s) relevant to the equipment involved.

Deliveries of new triaryl phosphate ester fluid should be supplied with a certificate of analysis/conformity. However, a delivery should be inspected to guard against the supply of an incorrect product. It is also advisable to sample the consignment and to test it against the specification. See ISO 3170 for guidance on the number of samples required.

When fluid is supplied in drums, the tests should be performed on the mean sample. However, individual samples shall be retained until the analysis of the mean sample has been completed and found satisfactory.

If sampling suggests the material is outside specification, then further investigation will be required. ISO 4259 gives guidance on how to judge the quality of a product with regard to specification limits and test method precision. However, it should be remembered that if a drum is opened to remove a sample, this should be taken under clean and dry conditions and the drum immediately resealed. Priority should be given to the use of any drum which 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 pipe work, or at least from the tanker bottom valve manifold; the sampling flow rate should be set to yield the 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 discharged is subject to negotiations between the supplier and user. However, consideration will 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.

In general, the shelf life of fluid in sealed drums is up to 2 years and during that time it may very slowly deteriorate. Therefore, if material is kept in stock for such a period, it is important to analyse it against the new fluid specification before use. If the product is found to be slightly outside specification (e.g. on acidity), it may still be used, as adding a small quantity to a larger volume of used fluid would be unlikely to have a significant adverse effect on bulk fluid properties. However, if in doubt, consult the fluid supplier.

12.2 Acceptance tests for new fluid

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

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

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

[a] The precision of particulate level measurement by automatic counting methods is poor and this parameter can often lead to disputes. The parties involved should recognize the limitations of the test method and seek guidance on precision in order to identify if there are grounds for a complaint.

12.3 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 an appropriate 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 Clause 9.

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 testing will depend on the limits on used fluid quality specified by either the turbine builder or the end user. Normally this will include the methods mentioned in Table 2 with the possible addition of tests for mineral oil, chlorine content, foaming and resistivity. The results should be compared with any limits on used fluid quality from the turbine builder, etc. or, in their absence, the warning limits given in Table 3. Some deterioration in performance over that of the fresh fluid is to be expected. If doubt exists as to whether the fluid is in a satisfactory condition for continued use, consult the turbine manufacturer or fluid supplier.

13 Examination of used fluid

13.1 General

While essential for monitoring fluid performance, testing is time-consuming and expensive. Some tests will duplicate the information already available while others are more indicative of a long term trend in performance than of immediate concern. Yet others can be used intermittently or when problems arise because normally there is little change in this property.

Tests therefore fall into two categories:

- those that are performed on every sample and are usually undertaken by the power station laboratory, which are typically the same as those used for checking new fluid quality (see Table 2): with the exception of appearance, for which a sample may be examined every one or two weeks, the frequency of testing is usually monthly, although this can vary depending on system design, operating conditions or if problems are being encountered;
- those that are carried out less frequently, which are often more specialized evaluations and usually involve an external laboratory, possibly that of the fluid supplier or manufacturer.

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

Table 3 lists the tests most often involved and the frequency of testing likely to be found during “normal” operation. It also indicates the measures to be taken in the event that analysis suggests a value close to, or exceeding, a warning limit.

Table 3 — Laboratory tests for inspection of triaryl phosphate esters fluids in service

Test	Warning limit	Frequency	Interpretation	Action steps
Appearance/Colour Visual/ISO 2049		Every month	For information on appearance, see 10.3 A slow darkening of the oil, while a sign of deterioration, is normal. A rapid darkening is not.	See 10.3 When colour is >4 and acidity difficult to control at a low value, treatment with ion exchange resins may assist. Contact the filter manufacturer.
Acid number (mg KOH/g) ISO 6619	0,3	Every month	Hydrolysis or oxidation taking place.	Switch to a treatment with an ion exchange resin.
	0,5			Consider a fluid change. (Above a 0,4 mg KOH/g acid number, control by fuller's earth treatment is not normally effective.)
Acid number increase between consecutive analyses (mg KOH/g) ISO 6619	0,1	Every month	Hydrolysis or oxidation taking place.	Change the solids treatment cartridges. Check water levels and dry fluid if necessary. Take samples every 48 h until return to normal.
Water content (% m/m) ISO 760/ISO 20764	0,1	Every month	Contamination.	Check air breather and heat exchangers.
Cleanliness[a] ISO 11500/ISO 4406	-/15/12 max	Every month	External (via tank) or internal contamination (fluid degradation/wear/filter malfunction, etc.).	Check filtration system, tightness of reservoir covers; whether breather fitted correctly.
Mineral oil content (%)	0,5 max	Every 3 months	Contamination.	Consider fluid change.
Mineral oil content - increase between consecutive analyses (%)	0,2	Every 3 months	Contamination.	Review top-up procedures and possibility of cross contamination at valves.
Metal content (mg/kg) ASTM D5185/ ASTM D6595	10 max	Every 3 months	Contamination, corrosion, wear and adsorbent solids	Identify source and remove, e.g. by resin treatment, fine particle filtration, etc.
Resistivity at 20°C (MΩ.m) IEC 60247	50 max	Every 3 months	Contamination (water/dirt), fluid ageing (acid).	Check top-up fluid. Correct water leakage. Change adsorption filters and take samples every 48 h until return to normal.
Air release at 50 °C ISO 9120	10 max	Every 6 months	Contamination or fluid ageing.	Investigate and correct any contamination. Consider fluid change.
Kinematic viscosity at 40 °C (mm^2/s) ISO 3104	$< \pm 5$ % of the initial value	Every 6 months	Cross contamination by a fluid of either lower or higher viscosity.	Check nature of contaminant and reduce/eliminate if possible. Identify source and/or ensure top-up procedure is correct. If contaminant cannot be removed, consider fluid change.

Table 3 (*continued*)

Test	Warning limit	Frequency	Interpretation	Action steps
Chlorine content (mg/kg) EN 14077	50 max	Every 6 months	Contamination.	If sea water contamination is suspected, check leaks; change adsorption filters and take samples every 48 h until return to normal.
Foaming ISO 6247, Sequence I at 24 °C (ml)	200/0 max	Every 12 months	Contamination – ageing.	Check for presence of foam in the tank. Check contamination by dirt, water, metal soaps and correct.
[a] Cleanliness testing frequency and limit will depend on the system pressure and the presence of servo-valves.				

13.2 Trend analysis

When analytical data has been obtained, it should be used to assess the current condition of the fluid and to decide if any corrective action is necessary. However, it can also be used to monitor trends in fluid behaviour, identifying what is normal and abnormal. It can also help predict the rate of degradation and when action may have to be taken; for example when adsorbent solid cartridges may have to be changed. This is particularly important in anticipating the condition of the fluid at the time of a scheduled outage as it may be appropriate to arrange a full or partial replacement at this time.

In the event that the test results are completely unexpected (but repeatable), or possibly conflict with other data, investigate the cause and, if necessary, contact the equipment manufacturer or fluid supplier, as they may be indicative of a system-related or fluid-related problem.

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