



912 Edition 12/2011

Quenchants – Monitoring and Maintenance





Technical Information 912 Edition 12/2011 Quenchants – Monitoring and Maintenance

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1. Quenchants – Definition

Hardening is the heat treatment of steel by the austeniting and subsequent cooling of the material's crystalline structure which leads to an increase in the hardness of the more or less fully realigned crystalline structure. Most often, the hardest martensite structure is required. However, some special applications specify a microstructure such as bainite.

The principal function of a quenchant is to dissipate the heat from the material at the speed required to create a crystalline structure which generates the hardness required. Secondary functions include corrosion protection for the machines and materials, good foaming characteristics, low evaporation and misting, good skin compatibility, a high flash point and good stability.

2. Storage of Quenchants

2.1. Quenching oils

Quenching oils can be stored for up to three years without detrimental effects on quality. However, they should be stored at temperatures between 5 and max. 40 °C. If drums are stored outdoors, they should be placed horizontally or at least suitably covered to avoid the ingress of water or moisture through the drum bungs.

2.2. Water-miscible, polymer quenchants

As a rule, water-miscible, polymer quenchants should only be stored for a maximum of one year. The containers should be clean and sealed and protected against the ingress of water or moisture as described in Section 2.1. Storage temperature must not fall below 5 °C and not exceed 40 °C. If storage tanks are used for watermiscible, polymer quenchants, they should be regularly checked for cleanliness and cleaned if necessary. We recommend that polymer quenchants are stored in stainless steel tanks.

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3. Application of water-miscible, polymer quenchants

3.1. Mixing rules

Water-miscible, polymer quenchant concentrates should be diluted to between 5 and 20% with water depending on the material-specific requirements. The following should be observed when diluting the concentrates:

3.1.1. Mixing water

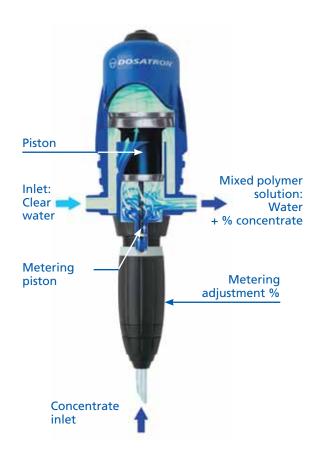
The quality of the mixing water is of decisive importance for the characteristics of a polymer solution. Firstly and in line with TRGS 611 (German Technical Regulations on Hazardous Substances), the water should have a nitrate content of less than 50 mg/l. If drinking water is used, this value will be given. Water hardness in ppm CaCO₃ determines the foaming behaviour of a polymer solution. If under 145 ppm CaCO₃, a polymer solution can tend to foam. If the water hardness is significantly over 360 ppm CaCO₃, calcium soaps can form, corrosion protection deteriorates, stability is lower and salts can precipitate on machine elements after longer use. The optimum water hardness is between 180 and 270 ppm CaCO₃.

The hardness of too soft water can be adjusted by the addition of "hardeners" such as calcium acetate and too hard water can be softened by adding fully demineralized water. The chloride content of the water should not exceed 50 mg/l because excess concentrations can lead to corrosion on machine elements and workpieces. Analyses of the water used can be obtained from the water supplier. If well water which is not subject to drinking water regulations is used, first check that the bacteria count is under 10³ because otherwise a greater risk of bacteriological contamination may be given. The temperature of the water used for dilution should under no circumstances be lower than 10°C because this can cause mixing problems.

3.1.2. Mixing of water-miscible polymer quenchants

When polymer quenching concentrates are diluted, the polymer concentrate should be added to the water until the required concentration is achieved. Smaller quantities can be mixed in a clean, separate container. The concentration of the solution should be measured with a refractometer. Optimally, larger quantities of polymer solutions should be mixed with dedicated mixing machines.

Apart from propeller and turbine stirrers, automatic mixing machinery is particularly recommended. Such equipment can be rigidly installed or fitted to drums or the tank. The connection to the drinking water supply must be fitted with a non-return valve to stop concentrate contaminating the drinking water supply (DIN 1988, Part 4). Even if the mixing apparatus can be adjusted to a specific concentration, the actual value should be checked with a refractometer and the product-specific refractometer factor and corrected if necessary. These factors are given in the corresponding Product Information sheets.



4. Monitoring Quenchants

4.1. Water-miscible, polymer quenchants

A number of disrupting factors can alter the application-specific properties of water-miscible, polymer quenchants. The quenchant should be routinely monitored to maintain maximum effectiveness and to keep potential health and environmental hazards in check. TRGS 611, which principally deals to watermiscible metalworking fluids, can be consulted.

The checks and the resulting measures should be coordinated with the quenchant manufacturer. To maintain economy and to keep costs down, it is imperative that characteristics offered by the product are retained for as long as possible. Apart from the quality of the product itself, service life largely depends on the quenching process and the scope and continuity of monitoring and maintenance measures.

These days with the use of central systems, it is simpler to maintain economic service life than with individually filled quenching baths. However, even the operators of individually filled systems can achieve astonishingly acceptable service life with the strict adherence to the quenchant manufacturer's and professional association's recommendations. Monitoring and maintenance measures during operation, i.e. the analysis of the polymer solution's condition and the use of maintenance apparatus, are of enormous importance. This is not just the case in terms of service life optimization. Users are also required to keep quenchants in good condition to fulfil safety-at-work regulations. Accordingly, operators have to create a monitoring plan and document the measured values and maintenance measures.

Analysis	Method	Recommended frequency
Appearance and odour	Visually and sensory	Daily
pH value	Electrometrically, DIN 51 369, pH strips	Every week – Recommended by TRGS 611
Polymer concentration	Refractometer, viscosity	Min. every week
Quenching characteristics IVF	FLP-A-18*	As necessary
Nitrite content	Test strips, photometry	Every week – Recommended by TRGS 611
Bacteria and count, fungus, yeasts	Dip Slide method	If necessary every week
Chloride content	Titration DIN 38 40 Part 9	As necessary
Water hardness	Ca, Mg with ICP	As necessary
Corrosion	Chip-filter test, DIN 51 360 Part 2	As necessary
Tramp oil, non-emulsified oil	In line with DIN 51 367	As necessary
Electrolyte content	Conductivity test	As necessary

4.1.2. Test methods for water-miscible, polymer quenchants

4.1.2.1 On-site testing

This section describes some simple on-site test methods:

• Visual checks

Two important checks which should be performed every day are in the foreground. The first is a basis requirement for perfectly functioning quenchants and concerns checking the **fluid level in the quenching bath.** Insufficient fluid can interfere with the circulation pump which can draw air and cause foaming.

This, in turn, causes further problems such as inadequate heat dissipation and unreliable quenching quality. Furthermore, appearance and odour should be checked every day. If visual or sensory alterations to the quenchant are noticed, these can be indicators of a change to the quenching fluid. These also require specific countermeasures along with a comprehensive analysis of the causes. In normal circumstances the solution should not appear oily or dark. A reddish-brown appearance and a slightly burnt odour is however normal and are the consequences of the heat treatment process. Changes to the quenchant can have a variety of causes and are mostly identified by the following monitoring parameters.

• pH value measurement

A pH value measurement should be performed once a week. The simplest way is to use test strips which show the current pH value by changing colour. As with all test strips, make sure that the best-by date has not passed. The pH value reading can be inaccurate if the strips are too old. If this method is used, the correct procedure must be followed. This means that the strip must be dipped into clean quenchant and not through floating tramp oil because this can also cause incorrect readings.

Furthermore, when the strips colour is evaluated, the time after which the reading is taken must be as the instructions dictate. The advantage of such a base value test is its speed and its simplicity which requires no additional chemicals. The simplicity of test strips also eliminates erroneous readings caused by incorrect use.



An alternative to the test strip method and somewhat exacter by more expensive is the use of electrical pH measuring apparatus. Regardless of whether a batterypowered, hand-held tester or a laboratory tester, it is important that the pH value electrodes are handled carefully and that the apparatus is routinely recalibrated.

Again, care must be taken that the pH electrodes in the semi-permeable membrane head are not passed through floating tramp oils for the previously-mentioned reasons.

The key to performing pH value measurements and their documentation is to illustrate development of the pH value against the service life of the polymer solution so that correction measures can be taken in good time.



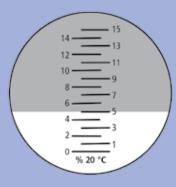
Concentration measurement

A concentration measurement should be performed at least once a week or, if necessary, daily if the quenching bath is extremely small or intensively used with high drag-out rates. A number of very simple and cost-effective methods are available. A concentration measuring tool which every heat treatment company should have is a hand-held refractometer.

• Hand-held refractometer

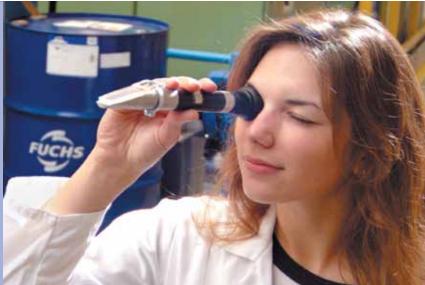
This is an investment of about EUR 200. By using the product-specific refractometer factor (given in the Product Information literature), concentration is measured similarly to the way sugar content (Brix value) in wine is determined by registering the refraction of light through the medium being measured. This can be seen by the separation of a dark greyish and a bright, clear zone. The value measured must then only be multiplied by the corresponding refractometer factor to give the concentration of the polymer solution.

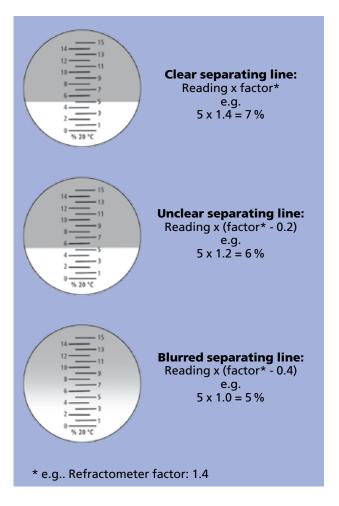
It is important to accurately set the zero line with pure water before any tests are carried out. A dirty, insufficiently stable or tramp oil-contaminated solution blurs the separation between light and dark and can lead to inaccurate readings.



Concentration = Reading x Refractometer factor

Reading: 5.0 Refractometer factor: 1.4 Concentration: 7.0 %





Digital hand-held refractometer

As opposed to a traditional hand-held refractometer, a digital hand-held refractometer is a battery-powered optoelectronic device. Only a few drops of the fluid are required to determine its concentration. The device shows a value after a few seconds and this again has to be multiplied by the product- or condition-specific factor to give the polymer concentration.



Note:

Although using a refractometer is a fast method of testing the concentration of polymer quenching fluids, the necessary correction factor changes with increasing product life.

The factors given in Product Information literature only apply to fresh mixtures. For this reason it is imperative that the concentration of the quenchant is tested viscosimetrically or by a test laboratory at certain intervals.

Kinematic viscosity

Regularly measuring the kinematic viscosity at 20 °C is a more reliable and accurate method of determining concentration. In addition, this measurement of viscosity can also generate an up-to-date multiplying factor for refractometer measurements. The combination of a quick refractometer test and the determination of the current correction factor by viscosity measurement offers a way of minimizing any possible fluctuations in concentration caused by ageing. The intervals at which viscosity measurements should be performed depends on all the influences a polymer solution is subjected to.



Principle of measuring kinematic viscosity according to DIN 51562-1

Kinematic viscosity is determined by the time required for a polymer solution to pass through a Ubbelohde capillary tube. Multiplying by the capillary constant gives the kinematic viscosity. Kinematic viscosity can be measured by a test laboratory or by the operator himself. Regular concentration measurements are recommended for intensively-used or small quenching baths.

In such cases, measurements performed by the operator are particularly useful and concentration testing kits are available on request.



Using a product-specific calibration function, the actual polymer concentration can be determined from the measured viscosity. The correction factor to calculate concentration from the hand-held refractometer value is generated as follows:

$v = k \star t$

- *ν*: Kinematic viscosity
- t: Time
- k: Capillary constant

 $Factor = \frac{Viscosimetric concentration factor}{Hand-held refractometer reading}$

• Quenching characteristics

Further to monitoring concentration by measuring viscosity and using a held-held refractometer, particularly large, intensively-used heat treatment baths should have their quenching characteristics checked a routine intervals. Recording the cooling curves shows if the concentration offers the quenching characteristics required. This can also offer an early warning of possible fluid ageing and countermeasures such as adding concentrate can be taken. An additional benefit is that service life can be significantly increased.



The applicable threshold value of < 20 ppm nitrite for metalworking fluids is soon reached in heat treatment applications. As a result, secondary amines are principally not used in our polymer quenchant concentrates and the inclusion of inhibitors eliminate the formation of nitrosamines. In these cases, TRGS 611 allows the stated threshold value to be exceeded.

Note:

Without doubt, the formation of nitroso-diethanolamine and the resulting MAK value* in metalworking fluids should be viewed critically. However, at present there is no accepted evaluation of nitrite content in polymer heat treatment quenchants. We therefore recommend contacting the corresponding Professional Associations if questions arise.

As nitrite, as previously mentioned, can originate from the nitrate in mixing water, the nitrate content should be checked at regular intervals. Corresponding test strips are available for such tests. According to drinking water regulations, a maximum of 50 ppm nitrate are permissible. As a rule, nitrate values are down in the 10-20 ppm range. Significantly high concentrations than 20 ppm are possible in heavily cultivated agricultural areas.

Nitrate tests need not be performed once a week. However, it is recommended to perform and document such measurements at least every six months or alternatively these values can be obtained from the local water supplier. Apart from the simple visual evaluation of test strips, it is also possible to use a so-called "Reflectoquant" which automatically reads the test strips. Such apparatus could be of interest if the operator has a large number of quenching baths to monitor.

> * MAK- value (maximum concentration value at the workplace)

Nitrite content (Nitrate content)

Although not mandatory for polymer quenchants but nevertheless useful for orientation purposes, the nitrite content of the fluid should be tested in line with TRGS 611. This states that the nitrite content should be routinely checked to avoid health hazards caused by nitrosamine contamination. Nitrite is a reactive component which, together with secondary amines, can form carcinogenic nitrosamine. Nitrite can enter quenchant systems through the use of nitrite-contaminated mixing water. Nitrite and nitrate are not contained in the formulation in polymer quenching concentrates.



An additional check which may be of benefit for individually filled baths with large top-up requirements but not mandatory, is the measurement of water hardness. This may be the case for mixing water which is drawn from the company's on-site well.

• Water Hardness

Here again it is possible to monitor the quenchant for increasing water hardness with simple-to-use and economical water hardness test strips. High evaporation rates can result is very hard water values.

The focus of current research work is the influence on heat dissipation. However, it is accepted that today's polymer solutions can cope with a high degree of water hardness. More of a problem is that hard waterrelated deposits and gumming can occur in quenching baths and nozzle systems which generate considerable cleaning costs. In general however, an increase in the hardness of the water is of detriment to the service life of polymer solutions.

A far more serious problem is the influence of water hardness on corrosion protection which may become inadequate and if unintentional over-dilution occurs can cause cost-intensive machine maintenance and down-time. This applies particularly to intensively or continuously used lines. Overall water hardness monitoring and the additional of fully demineralized water can eliminate a source of problems and can increase the service life of polymer solutions. In the past, three different test strips are needed to monitor pH value, nitrite content and overall water hardness. These days a so-called combitest is commercially available which measures all three parameters with just one strip.



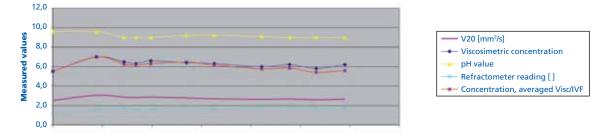
4.1.2.2. Monitoring schedule/Documentation

All monitoring work should be based on a monitoring schedule which details the values to be checked, the test methods, the test intervals, corresponding measures and any relevant product-specific information. An example is included below.

It is possible to add the measured values to lists or to generate a graphic record. The latter allows developments and trends in the polymer solutions to be seen at a glance. The values to be monitored and the corresponding intervals are plant-specific and should be discussed and determined in advance.

Quenching bath / Customer name											
Capacity [kg or litre]	100,000										
Recommended concentration [%]		6-7									
THERMISOL QZS 150 MM concentrate [kg]	tch 1										
Added water	Fresh batch Sample 1										
Date when water added	Fres										
Total added TH QS 150 MM											
Sample number	1	2	3	4	5	6	7	8	9	10	11
Date when sample taken	03.02.09	19.03.09	16.04.09	28.04.09	13.05.09	18.06.09	16.07.09	02.09.09	30.09.09	27.10.09	25.11.09
V20 [mm²/s]	2.5	3.0	2.9	2.8	2.9	2.8	2.7	2.6	2.7	2.6	2.7
Viscosimetric concentration	5.5	7.0	6.5	6.3	6.6	6.4	6.3	6.0	6.2	5.8	6.2
Concentration, IVF	5.5	7.5	6.0	6.0	6.0	6.5	6.0	5.5	5.5	5.0	5.0
Concentration, averaged Visc/IVF	5.5	7.0	6.3	6.2	6.3	6.5	6.2	5.8	5.9	5.4	5.6
Refractometer reading []	1.2	1.6	1.7	1.6	1.6	1.8	1.6	1.8	1.8	1.7	1.7
Corrected refractometer factor	4.6	4.4	3.7	3.8	3.9	3.6	3.8	3.2	3.3	3.2	3.3
pH value	9.6	9.6	9	9	9	9.2	9.2	9.1	9.0	9.0	9.0
Bacteria [10* 10x]	-	-	-	-	-	-	-	-	-	-	-
Yeasts											

THERMISOL QZS 150 MM



4.1.2.3. Maintenance options for water-miscible, polymer quenchants

Measured deviation	Possible consequence	Measure
pH value too low	Corrosion, instability	Use pH value-increasing additive
pH value to high	Nonferrous metal corrosion, skin compatibility problems	Check concentration and If necessary reduce Avoid the drag-in of alkaline cleaners
Water too hard	Deposits	Dilute top-fluid with fully de-mineralized water
Concentration too high	Slower quenching, lower hardness down to bainitic and pearlitic values	Reduce concentration by adding water
Concentration too low	Instability, abrupt quenching, cracking, quality problems	Add polymer concentrate until the desired concentration is achieved
Chloride concentration too high	Corrosion problems	Add demineralized water or stop drag-in on components
Bacteria contamination too high	Odour, pH value drop, skin compatibility problems	Expert addition of suitable biocide after contacting the manufacturer
Yeast contamination	Filtration problems, blocked circuits	Expert addition of suitable fungicide after contacting the manufacturer.
Nitrite content too high	Quenchant change	Eliminate source of contamination, e.g. quenching salts, add water, partial exchange of the quenchant
Conductivity too high	Instability corrosion	Find cause: Water too hard, magnesium? Drag-in of quenching salts adjust with demineralized water
Contamination too high	Poor quenching results	Improve filtration, remove scale contamination

4.1.2.4. Frequent practical problems with water-miscible polymer quenching concentrates – Causes and remedies

Foaming – Causes							
Water too soft	Excess aeration	Microbiological decomposition	De-foamers dragged- out	Dragged-in tramp oils	Concentration too high		
Countermeasures							
Harden water	Check fluid level and pumps. Reduce circulation Bath too small	Routine ventilation and circulation. Check pH value and concentration	Add de-foamer	Remove tramp oils regularly	Dilute as necessary with water		

Unpleasant odours – Causes						
Heavy contamination of the polymer solutionLonger periods of downtimeInsufficient ventilation of the systemContamination with tras- hed food or cigarette buttsPH value too low						
Countermeasures						
Check and improve cleaning measures	Circulate and ventilate	Circulate and ventilate	Instruct employees	Add alkalizator to correct pH value		

Deposits in the system – Causes							
Dragged-in tramp oilsDrag-in of cleaners or corrosion preventivesInsufficient cleaningMicrobiological decompositi products, fungi							
Countermeasures	Countermeasures						
Remove tramp oils with skimmers or separators Eliminate cause Partial renewal of polymer solution		Remove deposits, improve filtration	Add biocide or fungicide with fresh fluid, system cleaner, mechanical cleaning				

4.2 Quenching oils

As opposed to water-miscible polymer quenchants, quenching oils have an almost unlimited service life is correctly maintained. And bacteriological contamination cannot occur because of the absence of water. The optimum bath temperature is between 60 and 90 °C. Quenching baths used for the heat treatment of tool steels can, for example, be used at temperatures of up to about 200 °C. Solid contaminants should be continuously removed by a filter system. A disadvantage of quenching oils is their low flexibility concerning quenching characteristics and possible fire hazards. In addition and dependant on product quality, oil quenching can generate vapours which employees are constantly exposed to. These can be kept to a minimum if low-evaporation quenching oils are always selected.

4.2.1. Test procedures for quenching oils

Analysis	Method	Information about
Appearance and odour	Visual and sensory	Contamination, dragged-in tramp oils
Viscosity	DIN 51 562	Dragged-in tramp oils Ageing
Water content	Karl Fischer	Ingress of water via coolers, etc.
Saponification number	DIN 51 559	Additive levels, tramp oils
Neutralization number	DIN 51 558	Ageing, additive level
Density	DIN 51 757	Dragged-in tramp oils
Quenching characteristics IVF	FLP-A-18*	Quenching properties, ageing
Metal content	ICP, RFA, AAS DIN 51 391	Additive level dissolved or solid contaminants
Flash point	DIN ISO 2592	Dragged-in tramp oils or solvents, ageing
Evaporation loss	DIN 51 581 Part 1 or Part 2	Evaporation losses, ageing, contamination
Colour	DIN ISO 2049	Ageing, contamination, dragged-in tramp oils

* FUCHS EUROPE SCHMIERSTOFFE GMBH test procedure

4.2.2. Frequent practical problems with quenching oil – causes and remedies

Flaming	Abrupt quenching	Changed quenching characteristics	Deposits / increased oil drag-out	Unpleasant odour		
Cause						
Water	Water	Ageing	Ageing / oil quality	Ageing/oil quality		
Countermeasures						
"Evaporation" of water		Partial replacement/replacement				

5. Disposal of Quenchants

5.1 Reasons for the disposal of quenching oils

With suitable monitoring and maintenance, quenching oils have a very long service life. Nevertheless, it may at times be necessary to dispose of the product.

The following list some general disposal reasons:

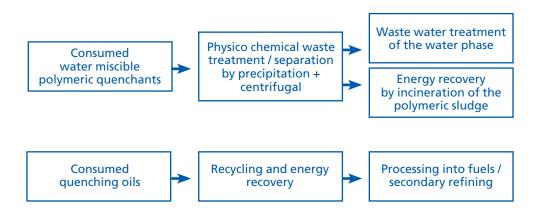
- Contamination with other lubricants
- Drag-in of cleaners or water-miscible emulsions
- Excess solid contaminants
- Oxidation ageing caused by high system temperatures.

5.2 Reasons for the disposal of water-miscible, polymer quenchants

Water-miscible polymer quenchant concentrates are diluted with water before use in quenching baths or inductive tempering lines. If the permissible condition criteria are no longer fulfilled during the fluid's use or for some other reason, the polymer quenching medium must be treated. Application criteria depend on the quenching process and the demands made on the quenchant. If these can no longer be fulfilled, the operator has to decide if the quenchant has reached the end of its useful life and has to be disposed of.

The following lists some criteria which can lead to disposal:

- pH value too low
- Water too hard
- Excess contamination with solids
- Quenching characteristics too abrupt
- Excess foaming
- Excess bacteria/yeast contamination
- Too much tramp oil
- Excess contamination with dragged-in substances
- etc.



5.3 Legislative provisions

The German waster oil directive deals with the classification of used and waste metalworking oils. Quenching oils are in Waste Oil category 2. Polymer Quenchants are grouped together with water-miscible metalworking fluids. Water-miscible metalworking fluids are excluded from the waste oil definition. Legislation on the implementation of the European Waste Directory views used and waste metalworking fluids as substances which need special attention. These metalworking fluids should be energetically recycled. As a result, the documentation accompanying metalworking fluid wastes must show the allocated Waste Codes. It must be pointed out that available technical information only gives generalized information on disposal and recycling. We recommend that operators contact local disposal companies for more details on the applicable legislation.

Types of Waste und Waste Codes

Waste	Examples	Waste Codes (EAV)
Halogen-free, mineral oil-based metalworking fluids	Used and waste quenching oils	120107 (Halogen free)
Synthetic metalworking fluids	Used and waste synthetic quenchants	120110
Halogen-free metalworking emulsions or solutions	Used and waste polymer quenchants	120109 (Halogen free)
Water-based, fluid wastes which contain hazardous substances	Used with barium chloride or cyanide contaminated polymer quenchants (Exception: if dragged-in from salt baths)	16 10 01

(European Waste Directory)

German references:

BGR 143 (bisher ZH 1/248) Regeln für Sicherheit und Umgangsschutz beim Umgang mit Kühlschmierstoffen; 1999

TRGS 611 Verwendungsbeschränkungen für wassermischbare bzw. wassergemischte Kühlschmierstoffe, bei deren Einsatz Nitrosamine entstehen können.

DIN 51385 - Schmierstoffe, Kühlschmierstoffe, Begriffe; 1991

DIN 51369 – Prüfung von Kühlschmierstoffen, Bestimmung des pH-Wertes des Kühlschmierstoffes; 1981

DIN 51360-2 – Prüfung von Kühlschmierstoffen, Bestimmung von Korrosionsschutzeigenschaften vom wassermischbaren Kühlschmierstoff; Späne/ Filterpapier Verfahren; 1981

VDI 3035 Anforderungen an Werkzeugmaschinen, Fertigungsanlagen und periphere Einrichtungen beim Einsatz von Kühlschmierstoffen; 1997

VDI 3397 Blatt 1 Kühlschmierstoffe für spanende und umformende Verfahren; 2007

VDI 3397 Blatt 2 Pflege von Kühlschmierstoffen für die Metallbe- und Verarbeitung. Maßnahmen zur Qualitätserhaltung, Abfall und Abwasserverminderung; 2005

VDI 3397 Blatt 3 Entsorgung von Kühlschmierstoffen; 2008

BIA-Gefahrstoffseminar G2 Praxisorientierte Anwendung der TRGS 611 Dipl.-Chem. Michael Roder, SM-BG, Mainz

BGHM – "Gefahren beim Betrieb von ausgewählten Abschreckbädern in der Härterei"; 2001

Betrieblicher Umweltschutz Baden-Württemberg – Metallbearbeitung und Galvanotechnik; 07.02.2011 http://www.umweltschutz-bw.de/index.php?lvl=351&timme=&oid=4187

Handbuch zum richtigen Umgang mit dem EAV 2001/118/EG – Reihe Abfall Heft 73 Band B Ministerium für Umwelt und Verkehr Baden-Württemberg

Your contact person

FUCHS EUROPE SCHMIERSTOFFE GMBH Friesenheimer Straße 19 68169 Mannheim / Germany Phone: +49 621 3701-0 Fax: +49 621 3701-570 E-mail: zentrale@fuchs-europe.de www.fuchs-europe.de

Export Division Friesenheimer Straße 19 68169 Mannheim / Germany Phone: +49 621 3701-1703 Fax: +49 621 3701-7719