

Iron LR L (B)

226

0.03 - 2 mg/l Fe

Ferrozine / Thioglycolate

## Instrument specific information

The test can be performed on the following devices. In addition, the required cuvette and the absorption range of the photometer are indicated.

Instrument Type	Cuvette	$\lambda$	Measuring Range
MD 600, MD 610, MD 640, XD 7000, XD 7500	ø 24 mm	560 nm	0.03 - 2 mg/l Fe

## Material

Required material (partly optional):

Reagents	Packaging Unit	Part Number
Iron LR 2 Reagent Set	1 Set	56R023490
KS135 Pa1/Alk1-Phenolphthalein Sub-Alk P	Liquid / 30 ml	56L013530
KS135 Pa1/Alk1-Phenolphthalein Sub-Alk P	Liquid / 65 ml	56L013565
KS135 Pa1/Alk1-Phenolphthalein Sub-Alk P	Liquid / 500 ml	56L013597
KS135 Pa1/Alk1-Phenolphthalein Sub-Alk P - pck of 5	Liquid / 1 ml	56L013572
KS144-CH2-FC4-Calcium Hardness Buffer	Liquid / 65 ml	56L014465
KS144-CH2-FC4-Calcium Hardness Buffer	Liquid / 1 ml	56L014472
KS144-CH2-FC4-Calcium Hardness	Liquid / 125 ml	56L014491

## Application List

- Cooling Water
- Boiler Water
- Galvanization
- Raw Water Treatment

## Preperation

1. If there are strong complexing agents in the sample, the response time must be extended until no further colour development is seen. However, very strong iron complexes are not included in the measurement. In this event, the complexing agent must be destroyed by means of oxidation with acid/persulphate and the sample also neutralised to pH 6–9.

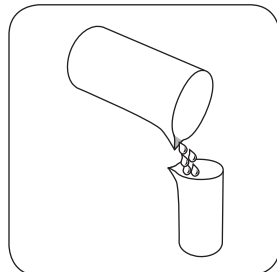
2. For the measurement of total iron, both suspended and dissolved, the sample must be boiled with acid/persulphate. It must be neutralised back to pH 6–9 and refilled to the original volume with deionised water.

## Notes

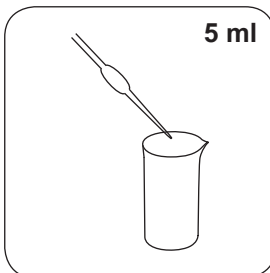
1. Do not add the reagent KS63 (Thioglycolate) if measuring  $\text{Fe}^{2+}$ .

## Digestion

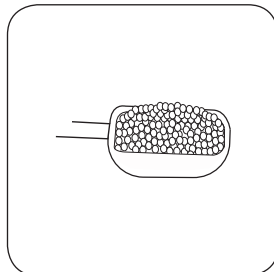
Total iron consists of suspended, soluble and complexed iron. The sample must be not filtered before measuring. To ensure homogenisation of the sample, deposited particles must be evenly distributed immediately prior to sampling by forcible shaking. A filtration of the sample is necessary for the determination of total soluble iron (including the complex iron compounds). The equipment required for the determination of total iron and reagents are not included in the standard delivery.



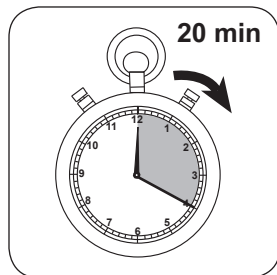
Fill a suitable digestion vessel with **50 ml homogenised sample**.



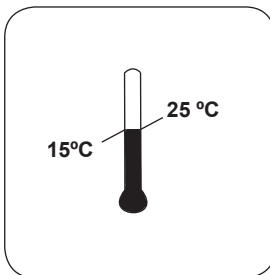
Add **5 ml 1:1 Hydrochloric acid**.



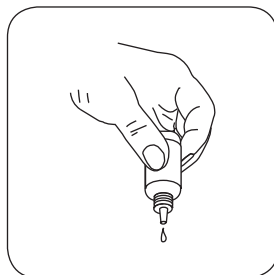
Add a measuring scoop **KP 962 (Ammonium Persulfat Powder)**.



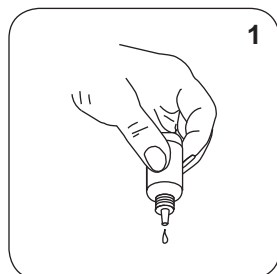
Boil the sample for **20 minutes**. A sample volume of about 25 ml should be retained; If necessary, fill with deionised water.



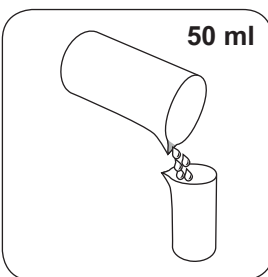
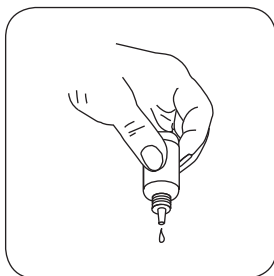
Allow the sample to cool to room temperature.



Hold cuvettes vertically and add equal drops by pressing slowly.



Add **1 drops KS135 (Phenolphthalein Substitute Indicator)**.



Add **KS 144 (Calcium Hardness Buffer)** drop by drop to the same sample until colouration turns from light pink to red. (**Note: make sure to swirl the vial after adding each drop!**)

Fill the sample with **deionised water to 50 ml**.

## Implementation of the provision Iron LR (B) with Liquid Reagent

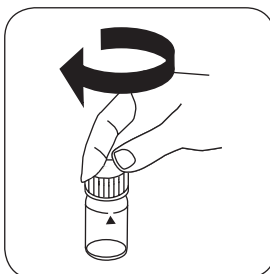
Select the method on the device

For this method, no ZERO measurements are to be carried out with the following devices: XD 7000, XD 7500

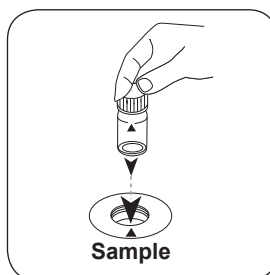
For determination of total dissolved iron with a distinction between  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  the sample must be filtered prior to the test (pore size 0,45  $\mu\text{m}$ ). Otherwise, iron particles and suspended iron are measured.



Fill 24 mm vial with **10 ml sample**.



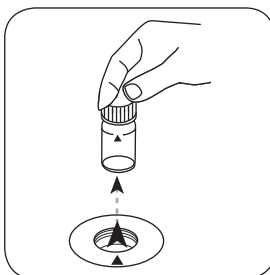
Close vial(s).



Place **sample vial** in the sample chamber. • Pay attention to the positioning.

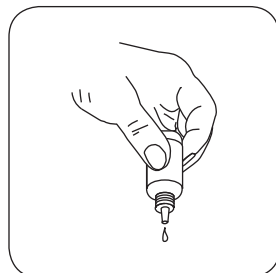


Press the **ZERO** button.

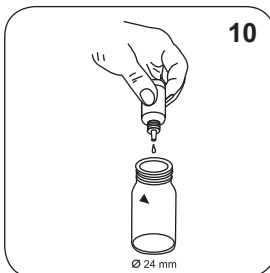


Remove the vial from the sample chamber.

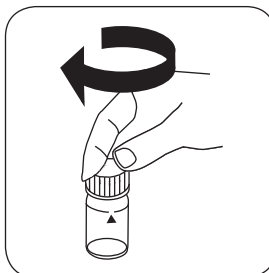
For devices that require **no ZERO measurement**, start here.



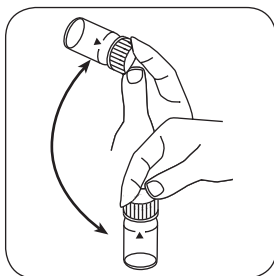
Hold cuvettes vertically and add equal drops by pressing **te Buffer** slowly.



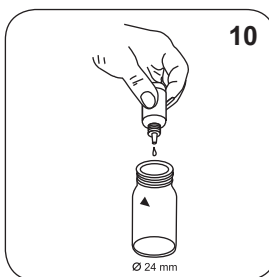
Add **10 drops KS60 (Acetate Buffer)**.



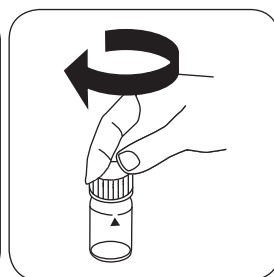
Close vial(s).



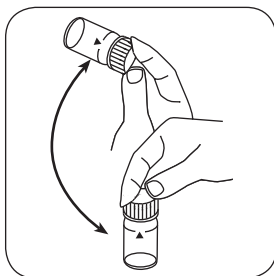
Invert several times to mix the contents.



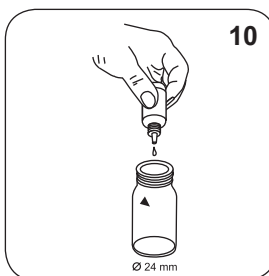
Add **10 drops KS63 (Thioglycolate)**.



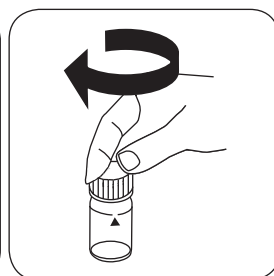
Close vial(s).



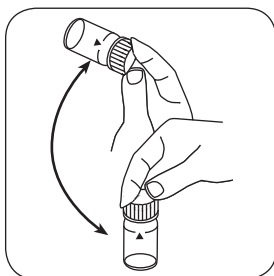
Invert several times to mix the contents.



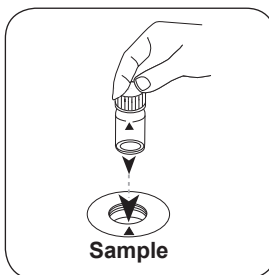
Add **10 drops KS65 (Ferrozine)**.



Close vial(s).



Invert several times to mix the contents.

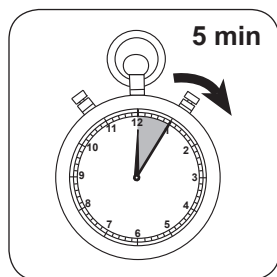


Place **sample vial** in the sample chamber. • Pay attention to the positioning.



Press the **TEST** (XD: **START**) button.

# Test



Wait for **5 minute(s) reaction time**.

Once the reaction period is finished, the measurement takes place automatically.

The result in mg/l  $\text{Fe}^{2+}/\text{Fe}^{3+}$ .  $\text{Fe}^{3+}=\text{Fe}^{2+}/3+-\text{Fe}^{2+}$  appears on the display.

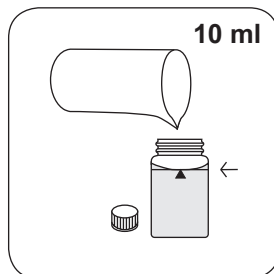
## Implementation of the provision Iron, total LR 2 with liquid re-agent

Select the method on the device

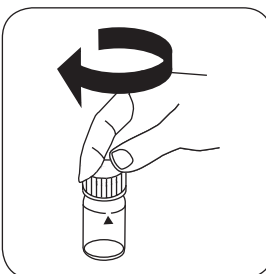
For testing of **Iron, total LR with liquid reagent**, carry out the described **digestion**.

For this method, no ZERO measurements are to be carried out with the following devices: XD 7000, XD 7500

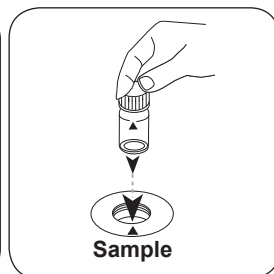
Total iron consists of suspended, soluble and complexed iron. The sample must be not filtered before measuring. To ensure homogenisation of the sample, deposited particles must be evenly distributed immediately prior to sampling by forcible shaking. A filtration of the sample is necessary for the determination of total soluble iron (including the complex iron compounds). The equipment required for the determination of total iron and reagents are not included in the standard delivery.



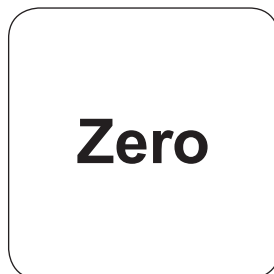
Fill 24 mm vial with **10 ml deionised water** .



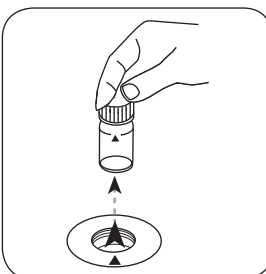
Close vial(s).



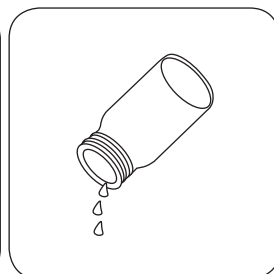
Place **sample vial** in the sample chamber. • Pay attention to the positioning.



Press the **ZERO** button.



Remove the vial from the sample chamber.



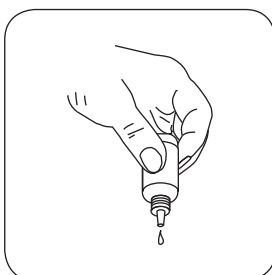
Empty vial.

For devices that require **no ZERO measurement** , start here.

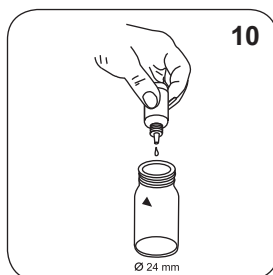




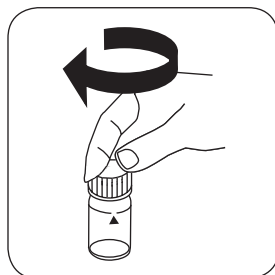
Fill 24 mm vial with **10 ml prepared sample**.



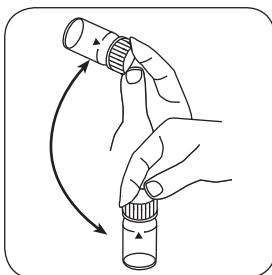
Hold cuvettes vertically and add equal drops by pressing slowly.



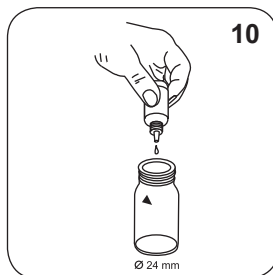
Add **10 drops KS60 (Acetate Buffer)**.



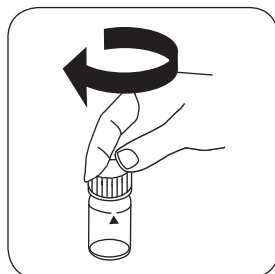
Close vial(s).



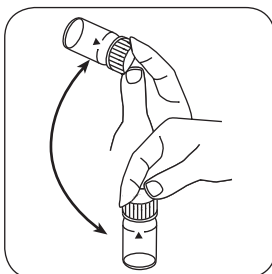
Invert several times to mix the contents.



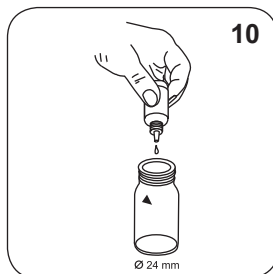
Add **10 drops KS63 (Thioglycolate)**.



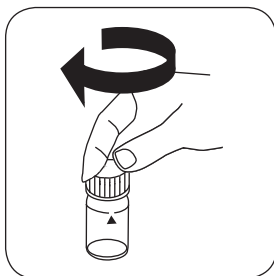
Close vial(s).



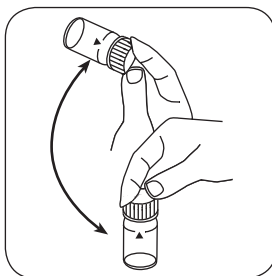
Invert several times to mix the contents.



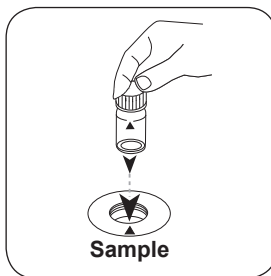
Add **10 drops KS65 (Ferrozine)**.



Close vial(s).



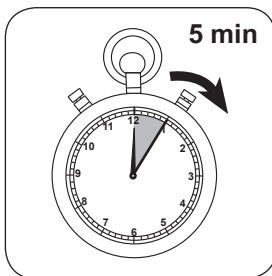
Invert several times to mix the contents.



Place **sample vial** in the sample chamber. • Pay attention to the positioning.



Press the **TEST** (XD: **START**) button.



Wait for **5 minute(s) reaction time**.

Once the reaction period is finished, the measurement takes place automatically.

The result in mg/l total Iron or when using a filtrated sample, in mg/l totale soluble Iron appears on the display.

## Chemical Method

Ferrozine / Thioglycolate

## Appendix

### Interferences

#### Removeable Interferences

- If using KS63 (Ferrozine/Thioglycolate), a high concentration of molybdate will result in an intense yellow colour. In this instance, a chemical blank value is required:
  - Use two clean 24 mm vials .
  - Mark one as blank for zeroing.
  - Fill a clean vial (24 mm) with 10 ml of the sample (blank).
  - Add **10 drops of KS63 (Thioglycolate)** to the vial.
  - Close the vial with the cap and swirl the contents to mix them.
  - Place the blank in the sample chamber. • Pay attention to the positioning.
  - Press the **ZERO** button.
  - Remove the vial from the sample chamber.
  - Fill a second clean vial (24 mm) with **10 ml of the sample** (this is the sample vial).
  - Add **10 drops of KS60 (Actate Buffer)** and as before, follow the procedure as described.

Interference	from / [mg/l]
Co	8
Cu	2
Oxalat	500
CN <sup>-</sup>	10
NO <sub>2</sub> <sup>-</sup>	

### Bibliography

D. F. Boltz and J. A. Howell, eds., Colorimetric Determination of Nonmetals, 2nd ed., Vol. 8, p. 304 (1978). Carpenter, J.F. „A New Field Method for Determining the Levels of Iron Contamination in Oilfield Completion Brine“, SPE International Symposium (2004)

<sup>a)</sup> determination of free, combined and total | <sup>b)</sup> Reactor is necessary for COD (150 °C), TOC (120 °C) and total -chromium, - phosphate, -nitrogen, (100 °C) | <sup>c)</sup> MultiDirect: Adapter is necessary for Vacu-vials® (Order code 19 20 75) | <sup>d)</sup> Spectroquant® is a Merck KGaA Trademark | <sup>e)</sup> alternative reagent, used instead of DPD No.1/No.3 in case of turbidity in the water sample caused by high concentration of calcium and/or high conductivity | <sup>f)</sup> additionally required for determination of bromine, chlorine dioxide and ozone in the presence of chlorine | <sup>g)</sup> Reagent recovers most insoluble iron oxides without digestion | <sup>h)</sup> additionally required for samples with hardness values above 300 mg/l CaCO<sub>3</sub> | <sup>i)</sup> high range by dilution | <sup>j)</sup> including stirring rod, 10 cm

