

REF 918 101

**Test 1-10**      **06.15**  
**NANOCOLOR® Lead**

en

**Extraction method****Method:**

Photometric determination with dithizone

Cuvette rectangular:	<b>50 mm</b>	<b>20 mm</b>	<b>10 mm</b>
Range (mg/L Pb <sup>2+</sup> ):	<b>0.005–0.500</b>	<b>0.02–1.00</b>	<b>0.03–1.00</b>
Wavelength (HW = 5–12 nm):	<b>520 nm</b>		
Reaction time:	<b>0</b>		
Reaction temperature:	<b>20–25 °C</b>		

**Contents of reagent set:**
**Box A:** 15 mL Lead R1  
 2 x 75 mL Lead R2  
 20 g Lead R4

**Box B:** 3 x 100 mL Lead R3  
 2 g wadding  
 2 measuring spoons 85 mm  
 10 g Lead R5

Additionally necessary is tetrachloroethylene p.a. or carbon tetrachloride p.a.

**Hazard warning:**

Reagent R1 contains ethanol solution 75–90 %. Reagent R2 contains ammonia solution 1–5 % Reagent R3 contains potassium cyanide solution 7–15 %. Reagent R4 contains hydroxylammonium chloride 80–100 %. H300, H310, H317, H330, H351, EUH032 Fatal if swallowed. Fatal in contact with skin. May cause an allergic skin reaction. Fatal if inhaled. Suspected of causing cancer. Contact with acids liberates very toxic gas. P201, P202, P260D, P262, P264W, P271, P272, P280sh, P301+310, P302+352, P304+340, P308+313, P330, P333+313, P361+364, P403+233, P405, P501 Obtain special instructions before use. Do not handle until all safety precautions have been read and understood. Do not breathe vapors. Do not get in eyes, on skin, or on clothing. Wash with water thoroughly after handling. Use only outdoors or in a well-ventilated area. Contaminated work clothing should not be allowed out of the workplace. Wear protective gloves/eye protection. IF SWALLOWED: Immediately call a POISON CENTER/doctor / ... IF ON SKIN: Wash with plenty of water / ... IF INHALED: Remove person to fresh air and keep comfortable for breathing. IF exposed or concerned: Get medical advice / attention. Rinse mouth. If skin irritation or rash occurs: Get medical advice / attention. Take off immediately all contaminated clothing and wash it before reuse. Store in a well-ventilated place. Keep container tightly closed. Store locked up. Dispose of contents / container to regulated waste treatment. For further information ask for a safety data sheet.

**Interferences:**The *total lead* can be determined with cracking set (REF 918 08).

By dilution of lead-containing samples the pH value must be stored lower than 3.

Interfering ions: Bi, In and Tl (0.4 mg/L Bi f 0.2 mg/L Pb). Other cations do not interfere below 10–100 mg/L. Phosphate ions in large concentrations inhibit the extraction. If sulphide ions are present, the test sample must be decomposed.

The method can not be applied for the analysis of sea water.

**Procedure (1<sup>st</sup> extraction):**
 Requisite accessories: 2 x 2 separation funnels 100 mL (REF 916 64), piston pipette with tips  
 Pour into two separate separation funnels:

Test sample	Blank value
<b>50 mL</b> test sample ( <i>the pH value of the sample must be between pH 1 and 3</i> )	<b>50 mL</b> distilled water
<b>5 drops</b> R1, mix (if solution turns blue, add diluted hydrochloric acid drop-by-drop until sample turns colourless)	<b>5 drops</b> R1, mix
<b>1 mL</b> R2, mix (sample turns blue, otherwise add more R2)	<b>1 mL</b> R2, mix (blank value turns blue)
<b>5 mL</b> R3, mix Add R4 in <b>small steps</b> while shaking until sample turns colourless.	<b>5 mL</b> R3, mix Add R4 in <b>small steps</b> while shaking until sample turns colourless.
<b>20 mL</b> organic phase <b>1 level spoon</b> R5, shake for <b>1 min</b> After phase separation use lower layer for 2 <sup>nd</sup> extraction, discard upper layer.	<b>20 mL</b> organic phase <b>1 level spoon</b> R5, shake for <b>1 min</b> After phase separation use lower layer for 2 <sup>nd</sup> extraction, discard upper layer.

**Procedure (2<sup>nd</sup> extraction):**

Pour into two other separate separation funnels:

Test sample	Blank value
<b>20 mL</b> distilled water add lower layer (organic phase) from 1 <sup>st</sup> extraction	<b>20 mL</b> distilled water add lower layer (organic phase) from 1 <sup>st</sup> extraction
<b>2 mL</b> R2	<b>2 mL</b> R2
<b>1 mL</b> R3, shake <b>1 min</b> , allow to separate	<b>1 mL</b> R3, shake <b>1 min</b> , allow to separate

After phase separation filter lower layers through funnels with wadding into two cuvettes and measure. Detoxify upper layers.

**Measurement:**

For NANOCOLOR® photometers see manual, test 1-10.

**Photometers of other manufacturers:**

Verify factor for each type of instrument by measuring standard solutions.

**Analytical quality control:**

NANOCOLOR Multistandard Metals 2 (REF 925 016)

**Detoxification:**

The aqueous phase must be treated with hydrogen peroxide (pH ca. 9), until no residual cyanide is detected.

**Disposal:**

Organic phase must be collect for waste disposal (chlorinated hydrocarbons). Please observe local regulations concerning disposal of waste.