

REF 918101

Test 1-10 12.23

NANOCOLOR® Lead

Extraction method

Method:

Photometric determination with dithizone

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

Contents of reagent set:

Box A: 15 mL Lead R1
2 × 75 mL Lead R2
20 g Lead R4

Box B: 3 × 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. as organic phase. This can be purchased from a chemical distributor, but not from MACHEREY-NAGEL.

Hazard warning:

Information regarding safety can be found on the box' label and in the safety data sheet. You can download the SDS from www.mn-net.com/SDS.

Interferences:

The *total* lead can be determined with cracking set (REF 91808).

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 (1st extraction):

Requisite accessories: 2 × 2 separation funnels 100 mL (REF 91664), piston pipette with tips

Pour into two separate separation funnels:

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

Procedure (2nd extraction):

Pour into two other separate separation funnels:

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

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

Measurement:

For MACHEREY-NAGEL photometers see manual, test 1-10.

Photometers of other manufacturers:

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

Analytical quality control:

NANOCONTROL Multistandard Metals 2 (REF 925016)

Detoxification:

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

Disposal:

Information regarding disposal can be found in the safety data sheet. You can download the SDS from www.mn-net.com/SDS.

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