REYM
Calle Ninos Heroes, Colas del Matamoros, Tijuana, Baja California, 22204, México
mchaides@reym.com.mx
http://reym.com.mx

TECHNICAL DATA SHEET

Zinni AL 450 S

Alkaline zinc nickel plating

Manufacturer	Atotech Deutschland GmbH
Document No.	1112
Version	18
Version date	25.11.2015
Doc.-No.	1112
SAP-No.	R: 1677995 B: 1670472
IMDS-No.	736126
Products covered	ZINNI AL 456 (1674852) ZINNI AL 457 (1674854) ZINNI AL 454 (1687826)

CONTENTS

1 Process Information

- **Zinni AL 450 S** produces semi-bright **zinc nickel** deposits with a consistent alloy composition of 12 –15 % nickel over the total current density and temperature ranges up to 30 °C.
- **Zinni AL 450 S** is designed for rack or barrel applications requiring a bendable deposit for post bending.
- **Zinni AL 450 S** has an excellent covering and throwing power as well as metal distribution.

2 Equipment

Anodes	**Nickel** anodes, or dull nickel plated (25 µm thickness) sheet steel anodes recommended. Area ratio of **anode to cathode** should be maintained in range **1 : (4 – 10**). **Do not use Nickel balls in titanium baskets or Bright Nickel plated steel sheet anodes**. **Zinc** dissolution takes place chemically through an externally mounted dissolving tank.
Tanks	Steel sheet tank lined with hard rubber, PVC, PVC/Polyester reinforced material, polyethylene or polypropylene. Plan steel tanks are satisfactory.
Pipes	Piping should be from alkaline resistant welded or threaded, reinforced polyester, polyethylene or polypropylene. **Glued PVC joints will fail and must not be used**
Cooling / Heating	Cooling is required to maintain a working solution temperature of 20 – 30 °C. Heat exchangers / heaters made from PTFE or titanium.
Exhaust	Required
Filtration	Required at 2 – 3 solution volumes per hour. Filter media: alkaline resistant, 10 – 20 µm rated.
Mechanical agitation	Slow cathode movement for rack (3 – 5 m/min) and rotation for barrel (3 – 8 rpm). When sufficient circulation is provided agitation of the cathodes is generally not necessary. **Never use air agitation**.

3 Make-Up

Make-Up of 100 l	Liter	kg
Water*	approx. 91	
NaOH		12.0
Zinc Oxide**		0.71
Zinni AL 456	1.0	
Zinni AL 457	4.7	
Zinni AL 454	0.15	

- * in areas of poor water quality, deionized water can be used.
- ** When using **Zinc** oxide it must be 99.9 % high purity grade with a max of 17 ppm Pb, max 3 ppm Cd, max 2 ppm Fe and 2 ppm Cu.
- Dissolve caustic soda in 1/5 of the required amount of water, if possible deionised water.
  Attention strong warming!!
- Then add to this still hot solution **zinc** oxide, stir well, and dissolve to a clear solution.
- Allow to cool down to room temperature, add up with water to 85 % and filtrate the bath if turbid.
- Please **note** that **Zinni AL 456 Ni** and **Zinni AL 457** must always be premixed before addition to the bath.
- Then add the required amount of **Zinni AL 454** additives and fill up the bath to desired volume with water.
- The electrolyte has to be circulated for approximately 24 hours before use.
- It is suggested to electrolyse the solution for several hours at normal current densities before starting regular plating.

Important Note: In all work with Zinni AL 450 S make sure that all general regulations on working with alkaline solutions are carefully observed!

4 Working Parameters

Temperature	25 °C (22 – 30 °C)
Voltage	2 – 6 V for rack plating 6 – 12 V for barrel plating Contacts from racks or barrels should cleaned regular.
Current density	Cathodic: 1.75 A/dm² (1.0 – 2.5 A/dm²) for rack plating For **fresh make-up** lower current densities have to be used 1.25 A/dm² (1.0 – 1.5 A/dm²) for rack plating **Anodic**: 3.0 – 10.0 A/dm² for rack plating

5 Maintenance

Nominal values	Make up g/l	Operation
Zinc metal	5.6	5.5 – 6.2
Nickel metal	0.7	0.7 – 2.0
Sodium Hydroxide	120	100 – 140

For fresh make-up Zinni AL 457 should not be higher than 4.5 liter on 100 liter to have Nickel incorporation below 15 %.

Sodium Carbonate

Sodium Carbonate naturally occurs in cyanide-free alkaline electrolytes and its content increases under normal operation. High levels of carbonate will reduce the plating efficiency of the electrolyte, so should be regularly monitored and kept below a maximum level of 50 g/l.

We recommend making frequent, small additions of all additives to insure constantly good results. If brightness of the work falls off rapidly, it is recommended to make a chemical analysis of the plating solution and bring the bath back to normal conditions before making any large additions of additives. The additives cannot compensate improperly balanced plating solutions.

	Consumption for 10,000 Ah
Zinni AL 456	9.0 – 13.0 l
Zinni AL 457	0.5 – 2.0 l
Zinni AL 454	1.0 – 2.0 l

***Note**: consumption rates are based upon practical experience and may need adjustment to reflect local operating conditions and substrate material types.*

Important Note: Zinni AL 456 and Zinni AL 457 must always be premixed before addition to the bath!

Function of the Additives

Zinni AL 456	**Nickel** replenishment additive (0.1 g/l Ni = 1.4 ml/l).
Zinni AL 457	Complexing additive required for proper bath performance.
Zinni AL 454	Main brightener.

6 Production Downtimes

After production downtimes the electrolyte has to be checked via hull cell test before production can be continued.

7 Guidelines for the Storage of Products of Zinni AL 450 S

Zinni AL 450 S products should be stored at normal indoor conditions (8 – 30 °C).

Do not store **Zinni AL 450 S** products for extended period at temperatures above 40 °C or below 0 °C. This can lead to a shorter shelf life or to freezing of the product.

8 Application Guidelines

Zinc:

The **zinc** has to be dissolved in a separate tank by using zinc anodes 99.99 % in iron or **nickel** plated iron baskets. The zinc solution will be added to the bath using a filter pump.

Nickel:

The content of **nickel** in the electrolyte will be adjusted by the nickel solution **Zinni AL 456**. It is recommended to control the nickel content in the electrolyte as well as in the deposit (AAS, X-Ray). For a good passivation ability and corrosion resistance it is important to maintain the nickel content in the deposit between 12 – 15 %.

The following parameters influence the **nickel** content in the deposit:

- The ratio Ni : Zn in the electrolyte.
- Higher content of **nickel** in the electrolyte increases the content of nickel in the deposit.
- Higher content of **zinc** in the electrolyte decreases the content of **nickel** in the deposit.
- All parameters which decrease the current efficiency for the **zinc** plating increase the percentage of **nickel** in the deposit.

These are:

- Lower **zinc** content.
- High carbonate content.
- Too low temperature.
- Overloaded on **Zinni AL 457 and/or Zinni AL 454**.

The electrolyte will be controlled by analysis and in hull cell.

9 General Safety Precautions

Avoid direct contact with this material. Do not inhale associated mist or vapors. Wash contaminated clothing before reuse. Refer to Material Safety Data Sheet (MSDS) for specific precautions before handling this material and for first aid recommendations. As applicable, keep exposure below the limits recommended by the appropriate regional regulatory agencies.

For further information on product safety refer to the corresponding MSDS. The MSDS is enclosed at the first delivery of a product. Otherwise a MSDS for a product is available on request from the appropriate regional Atotech office.

10 Zinc dissolving system

inert anodes	
zinc anodes	

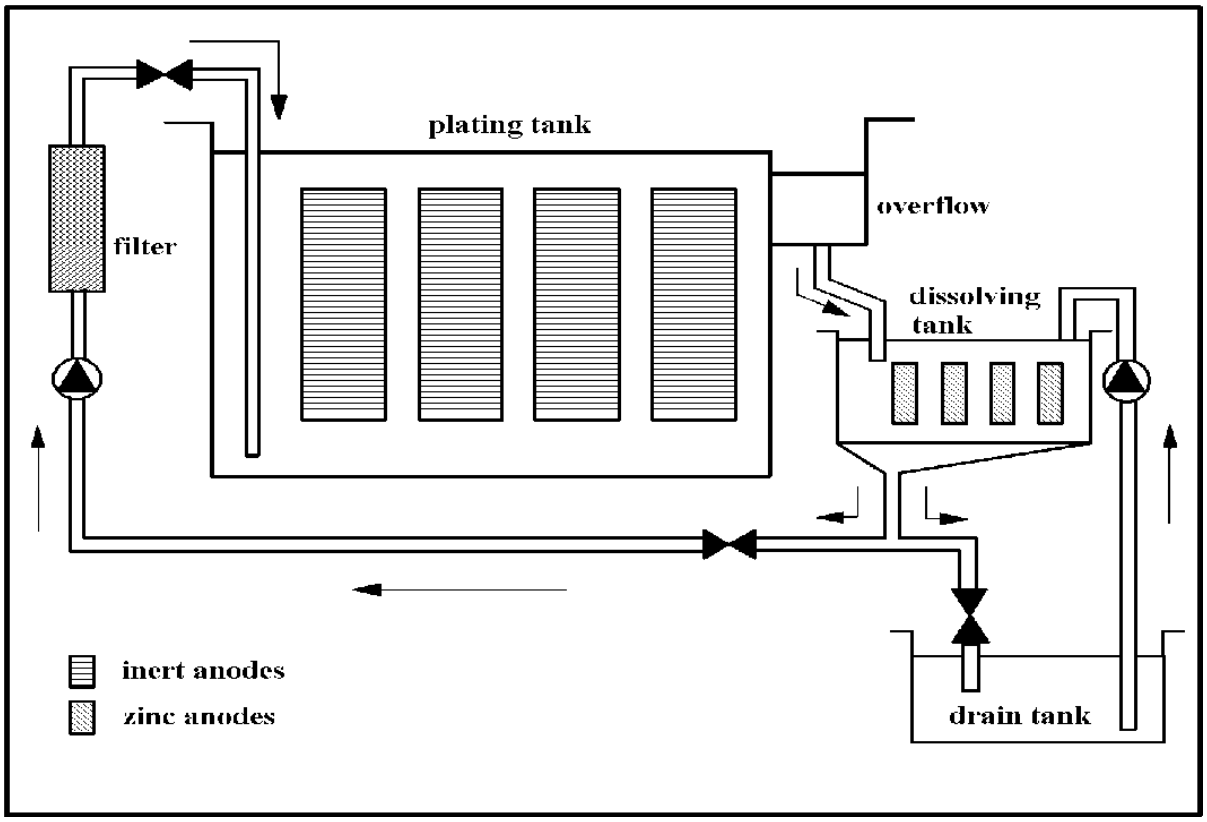

Figure from page 7 of the original document

Guidelines for Equipment and Operation

- Volume of the dissolving tank should be 10 – 20 % of the volume of the plating tank.
- Capacity of the filter pump should be 1 – 3 times per hour of the total bath volume.
- The filter unit must be installed between the dissolving and plating tanks (see drawing).
- **Zinc** metal should be placed into steel mesh baskets into the dissolving tank; zinc in contact with steel will dissolve at a faster rate.
- For optimum dissolution we recommend 2 cm diameter **zinc** balls to achieve sufficient zinc surface area.
- Anode baskets should be made from ST37 carbon steel mesh with a ratio of surface area to volume > 4 dm^{-1}.
- To ensure optimum activation of the steel mesh, it should be prepared by degreasing, then etching in dilute HCl followed by water rinsing.
- During start-up the **zinc** metal content of the electrolyte must be checked by analytical means. The zinc surface area under the solution in the dissolution tank can then be adjusted to maintain the correct zinc concentration in the operating tank, generally the quantity of zinc should be approximately 10 times greater than the daily consumption.
- If production operations are interrupted for relatively long periods of time, the solution should be pumped into the drain tank, or the **zinc** anode baskets should be removed, to prevent an increase in zinc concentration. After draining the zinc metal, the baskets should be cleaned by thoroughly water rinsing.
- An acceleration of **zinc** dissolution can occur when electrolyte replenishment additions of sodium hydroxide are added directly into the dissolution tank.

During none production periods it is essential that the zinc anode material is not left in prolonged contact with solution contained within the dissolution tank.

Zinc concentration in the dissolution tank must not exceed 20 g/l, as precipitation and deactivation of the basket material may occur. Under normal operation this will be maintained by adequate electrolyte exchange between the process tank and the dissolution tank.

Do not make any chemical additions directly into the dissolution tank, it is recommended to use a separate mixing tank solely for additions.

11 SAP No. for Make - Up and Maintenance Products

PRODUCT	SAP No.
ZINNI AL 456	1674852
ZINNI AL 457	1674854
ZINNI AL 454	1687826

12 Recommendations for Wastewater Treatment

Testing has shown that a small amount of cyanide may be generated when plating with **Zinni AL 450 S** process. Facilities using the Zinni AL 450 S process should take precautions appropriate to the management of cyanide-containing materials.

The product contains complexing agents and must therefore be strictly kept separate from rinse water circulation plants.

Rinse water (about 300 mg/l total heavy metal content) and bath concentrates (diluted in a ratio of a at least 1 : 40 prior to treatment) can be treated by an oxidation followed by an organosulfide precipitation.

The diluted solution to be treated is alcalized to a pH of 12.5 by adding caustic soda solution. Afterwards, fresh sodium hypochlorite solution (NaOCl, 14 % active chlorine) is added (approx. 10 – 35 l/m³) until an excess of oxidization agent occurs, which can be noticed by the blue colour on the potassium iodide starch paper.

After a reaction time of about 4 h while stirring thoroughly at a constant pH of 12 the paper should have a weak blue colour.

The excess of oxidization agent can be removed – check with potassium iodide starch paper by adding a small amount of sodium dithionite powder ($Na_2S_2O_4$) approx. 1 – 1.5 kg/m³. After adjusting the pH to 6 with hydrochloric acid, now organosulfide solution Sediganth C is added, about 3.7 l/m³. The indication can also be carried out by potentiometrically.

The amount of Sediganth C (about 8 – 9 per 1 g heavy metal) has to be determined by a prior test (as described below). After a reaction time of about 30 min. a possible excess of organosulfide has to be removed by $FeCl_3$ solution. The pH is then adjusted to about 8.5 with calcium hydroxide (milk of lime) and the solution is then filtered over a special filter press after adding some flocculant agent.

The filtrate can then be sent to the metal free wastewaters for final neutralization bypassing the wastewater treatment plant.

To determine the required amount of precipitation agent:

- 0.1 ml precipitation agent Sediganth C is added to
- 100 ml of rinse water **sample** at a pH of 4 – 10 while stirring thoroughly.
- After a reaction time (while the solution is thoroughly stirred) of 5 min, the solution to be treated has to be clear and colourless.
- When another 0.1 ml precipitation agent Sediganth C is added no further clouding should appear.

The resulting amount of ml of Sediganth C used per 100 l water **sample** gives the required amount of Sediganth C in Liter for 100 l rinse water.

Alternatives for the treatment of concentrate and rinse water with higher concentrations:

For treatment of higher concentrated rinse waters and concentrates with we recommend to direct the collected wastewater to a special incineration plant. As an alternative, it is possible to treat the wastewaters by an oxidation via an UV/H_2O_2 plant and additionally e.g. in combination with a selective cation exchanger.

This recommendation for the treatment of wastewater is in accordance with the Regulation on requirements for the discharge of wastewater into water (Allgemeine Abwasserverordnung , AbwV) and was issued on the basis of the limits provided in Annex 40 of the AbwV " Metal Handling and Metal Processing".
Be sure to observe all local regulations concerning the limiting values of pollutants including the general regulations on dangerous materials!

13 Analytical Instructions

Determination of rel. Total Carrier in Zinni AL 450 S by Titration

Manufacturer's method · Version 01 · Doc.-No. EXT-1284-TIT-01

Reagents required:*

- 0.1 M sodium thiosulfate volumetric solution
- Starch solution 1 % in water (shortly boil!)
- Copper sulfate solution 5 %
- Sulfuric acid 10 %
- Potassium iodide 30 %
- **Fresh Make-up** of **Zinni AL 450 S**

Equipment required:

- Pipette 5 ml
- Burette 50 ml
- Beaker 250 ml
- Volumetric flask 100 ml
- 540 Filter paper

Please follow the general regulations for hazardous materials in the Material safety data sheets!
The analytical procedure is to be followed exactly.
Be sure to observe all local regulations concerning the permitted limits for pollutants.

Procedure:

- 5.0 ml ** solution will be pipetted into a 250 ml beaker. Then add
- 20 ml copper sulfate solution.
- The precipitated deposit is then filtered through 540 grade paper and rinsed thoroughly with DI water. To the collected and mixed filtrates add:
- 10 ml sulfuric acid. After addition of
- 20 ml potassium iodide solution carefully titrate with
- 0.1 M sodium thiosulfate volumetric solution until the color of the solution changes from brown to light-yellow. Now add 1 ml of starch solution and further titrate until the dark blue color of the solution changes to colorless.
- **a fresh bath make up is analyzed in the same way**.

Calculation:

(consumption of 0.1 M sodium thiosulfate volumetric solution*** used for bath **sample**) /
(consumption of 0.1 M sodium thiosulfate volumetric solution*** used for **fresh make up**) * 100%
is the **Rel. Total Carrier in** %

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.
- ** Volumes given exact to a decimal point must be measured with a volumetric pipette.
- *** For the purposes of **calculation**, the factor of the volumetric solutions is assumed to be 1.000 and is therefore not taken into account in the **example**s.

Determination of Nickel by AA

Manufacturer's method · Version 05 · Doc.-No. A0000170-05

Principle:
Atomic Absorption Spectrometry (F-AAS)

The **sample** containing **nickel** is diffused and after atomizing in a compression air/acetylene-flame of the flame-atomic absorption spectrometer, the absorption is measured at a wavelength of 232.0 nm. The determination is carried out through external calibration by using corresponding standard solution.

Reagents required*:

- **Nickel** stock solution (Cni = 1.000 g/l), Fa Kraft
- Multi element stock solution Merck IV(1000 mg/l)
- Nitric acid, conc. (HNO_3 65 %), Fa. Merck
- Hydrogen peroxide (30%)

- DI water (millipore quality)

Please follow the general regulations for hazardous materials in the Material safety data sheets!
The analytical procedure is to be followed exactly.
Be sure to observe all local regulations concerning the permitted limits for pollutants.

Equipment:

- Atomic Absorption Spectrometer with control and **evaluation** unit (e.g. Agilent AA280FS) or equivalent auto**sample**r
- Burner: Air/Acetylene one-slit burner head
- Hollow cathode lamp: Ni
- Standard laboratory **equipment**

The above listed equipment is only valid when this method was created. For purchase of new analytical equipment the recommend equipment list has to be followed:
https://10.187.127.52/Communication/html/scientific_services_chemical_services_analytics.php

Parameter for Agilent AA280FS:

Nickel (Ni)	
Operating mode	Extinction
Delay	15 s
Wavelength	232.0 nm
Rinse time	20 s
Integration time	1.0 s
Gas mixture	air/acetylene
Replicates	10
Fuel flow	2.0 L/min s
Background correction	Yes
Oxidant flow	13.5 L/min
Calibration	New-Rational
Slit	0.2 nm

Please consult the instruction manual provided by the manufacturer of the AAS system, for information on the individual parameters such as the flame, the slit width, the integration period, etc.

Procedure:

Calibration standards:

Ni-Standards	S_1 = 0.5 mg/l S_2 = 2 mg/l S_3 = 5 mg/l

Exactly 0.05** ml, 0.2 ml and 0.5 ml of the Ni stock solution are transferred into a 100 ml volumetric flask, 4 ml conc. HNO_3 are added and the tubes are filled up to mark with DI-water.

It is possible to use a Sample Introduction Pump System (SIPS). In this case use a Ni standard solution with a concentration of 5 mg/L (SIPS stabilization time: 7 s).

Reference solution

Ni-reference	Sref = 3.0 mg/l

Exactly 0.3 ml of the Multi element stock solution are transferred into a 100 ml volumetric flask, 4 ml conc. HNO_3 are added and the flask is filled up to mark with DI-water.

Sample Solutions

- 1.0 ml of the solution are placed into a 100 ml volumetric flask and mixed with
- 10 ml nitric acid conc. and

- 1 ml hydrogen peroxide.
- Now heat up the solution until all hydrogen peroxide is boiled out
- Afterwards allow the solution to cool down to RT and fill up to the mark with DI water.
- Dilute this solution again 1:5 with DI water (final dilution; 1: 500).

Evaluation:

The concentration of **Nickel** is evaluated by external calibration via the F-AAS software. The determined absorption must be within the calibrating curve, otherwise another dilution has to be chosen.

$$\frac{\text{Content Ni (mg/l) AAS}}{1000} \times \text{Dilution} = \text{Nickel Content (g/l)}$$

Example:

$$\frac{3.5 \text{ mg/l AAS}}{1000} \times 500 = 1.8 \text{ g/l Ni}$$

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.
- ** Volumes given exact to a decimal point must be measured with a volumetric pipette.

Gravimetric determination of Nickel in alkaline zinc baths

Manufacturer's method · Version 01 · Doc.-No. A0001852-01

Principle: Nickel shows a precipitation, which can be filtrated and weighed to calculate the amount of Nickel.

Reagents* required:

- DI water (at least 60°C and ambient temp.)
- Precipitating agent:
  - 0,1 M Dimetyhlglyoxim solution in water (30,4 g Dimethylglyoxim sodiumsalt octahydrate)
- Glacial acetic acid
- 5 M Ammonia solution
- 5 M Hydrochloric acid

Please follow the general regulations for hazardous materials in the Material safety data sheets!
The analytical procedure is to be followed exactly.
Be sure to observe all local regulations concerning the permitted limits for pollutants.

Equipment required:

- Usual laboratory **equipment**
- Rubber wiper (spade shade) on a glass rod
- Graduated burette 25.0 ml, class A or AS
- Vacuum filtration apparatus with filter flask
- Filter-crucible 30 ml p4, glass
- Multicolored pH-strips pH 0-14 and pH 7.5-14.0,
- Drying oven
- Desiccators
- Various pipettes

Preparation:

If the crucible(s) contains any residue, clear them with 5 M hydrochloric acid until it is dissolved.
Then wash them with DI-water until there are free of acid (check with pH-strips) by using the vaccum filtration.

Dry the crucible(s) in the oven at 115-120° C (239°-248° F) for at least 2h, cool it down in the desiccators and weight it correct to four decimal places, which gives you weight A in g.

Procedure:

Transfer 10.0 ml* **sample** (if an amount less then 2 g/l is expected take 20 ml) into a 150 ml Beaker and add about 20 ml of DI water

Decrease the pH-value of the **sample** to 8-9 by adding glacial acetic acid (1.8 ml should suffice). Check with ph-Strips (7.5-14.0). If the pH doesn't match add more glacial acetic acid carefully or if it is to low readjust with 5 M ammonia solution.

Cover the beaker with a watch glass dish and heat the **sample** until it boils.

Let it cool down for 2 min and add 25 ml precipitating agent by using the burette and shake the beaker carefully. The poorly soluble red complex should fall out immediately.

Keep the **sample** warm (at least 60°C) on the heater for 30 min, make sure it is covered with a watch glass dish.

Filter the fallout using the vacuum filtration with a filter flask and the dried crucible.

If residue remains in the beaker use the filtrate to transfer it.

Wipe the beaker with the rubber wiper, so remaining residue is collected at the bottom, use warm DI water to transfer it as well.

Wash the residue in the crucible with 10 ml warm DI water and 20 ml DI water at ambient temp.

Dry the crucible in the drying oven at 115-120° C (239°-248° F) for at least 2h, cool it down in the desiccators (15 min should suffice) and weight it correct to four decimal places.

Repeat this **procedure** with 30 min drying until the weight is steady, that gives you weight B in g.

Check the pH-value of the filtrate and readjust it to pH 8-9 if necessary using either glacial acetic acid or 5 M ammonia solution.

Add 1 ml precipitating agent and heat the solution up to 70°C. If fallout emerges, the reaction was incomplete.

If the pH-value of the filtrate was lower then 8, just keep the **sample** warm for another 30min and filtrate again with the crucible already used.

If the pH-value was high enough, repeat the assay from the beginning with a smaller **sample** volume.

Evaluation: The difference of weight B minus weight A multiplied with 20,317 (10.159) gives you the amount of **nickel** in the bath in g/l for a **sample** volume of 10 ml (20 ml).

Example: (30.2069 – 29.9917) x 20.317 = 0.2152 x 20.317 = 4.4 g/l

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.
- ** Volumes given exact to a decimal point must be measured with a volumetric pipette.

Determination of Zinc by F-AAS

Manufacturer's method · Version 04 · Doc.-No. A0000171-04

Principle:
Atomic Absorption Spectrometry (F-AAS)

The **sample** containing **zinc** is diffused and after atomizing in a compression air/acetylene-flame of the flame-atomic absorption spectrometer, the absorption is measured at a wavelength of 213.8 nm. The determination is carried out through external calibration by using corresponding standard solution.

Reagents required*:

- Zink stock solution (cNi = 1.000 g/l), Fa. Kraft
- Multi element stock solution Merck IV(1000 mg/l)
- Nitric acid, conc. (HNO_3 65 %), Fa. Merck
- Hydrogen peroxide (30%)
- DI water

Please follow the general regulations for hazardous materials in the Material safety data sheets!
The analytical procedure is to be followed exactly.
Be sure to observe all local regulations concerning the permitted limits for pollutants.

Equipment:

- Atomic Absorption Spectrometer with control and **evaluation** unit (e.g. Agilent AA280FS) or equivalent Auto**sample**r

- Burner: Air/Acetylene one-slit burner head
- Hollow cathode lamp: Zn
- Standard laboratory **equipment**

***The above listed equipment is only valid when this method was created. For purchase of new analytical equipment the recommend equipment list has to be followed**:*
https://10.187.127.52/Communication/html/scientific_services_chemical_services_analytics.php

Parameter for Agilent AA280FS:

Nickel (Ni)	
Operating mode	Extinction
Delay	15 s
Wavelength	213.8 nm
Rinse time	20 s
Integration time	1.0 s
Gas mixture	air/acetylene
Replicates	10
Fuel flow	2.0 L/min s
Background correction	Yes
Oxidant flow	13.5 L/min
Calibration	New-Rational
Slit	0.7 nm

***Please consult the instruction manual provided by the manufacturer of the AAS system, for information on the individual parameters such as the flame, the slit width, the integration period, etc. For different F-AAS systems the parameters have to be adjusted correspondingly**!*

Procedure:

Calibration standards:

Zn-Standards	S_1 = 0.2 mg/l S_2 = 0.5 mg/l S_3 = 1.0 mg/l

Exactly 0.02 ml, 0.05 ml and 0.1 ml of the Zink stock solution are transferred into a 100 ml volumetric flask, 4 ml conc. HNO_3 are added and the tubes are filled up to mark with DI-water.

It is possible to use a Sample Introduction Pump System (SIPS). In this case use a Zn standard solution with a concentration of 1 mg/L (SIPS stabilization time: 7 s).

Reference solution

Zn-reference	Sref = 0.8 mg/l

Exactly 0.08 ml of the Multi element IV stock solution are transferred into a 100 ml volumetric flask, 4 ml conc. HNO_3 are added and the flask is filled up to mark with DI-water.

Sample Solutions

- 1.0 ml** of the solution are placed into a 100 ml volumetric flask and mixed with
- 10 ml nitric acid conc. and
- 1 ml hydrogen peroxide.
- Now heat up the solution until all hydrogen peroxide is boiled out..
- Afterwards allow the solution to cool down to RT and fill up to the mark with DI water
- Dilute this solution again with 1/100 of DI water (final dilution: 1:10,000).

Evaluation:

The concentration of **zinc** is determined by external calibration via the F-AAS software. The determined absorption of the **sample** must be within the calibrating curve, otherwise another dilution has to be chosen.

$$\frac{\text{Content Zn (mg/l) AAS}}{1000} \times \text{Dilution} = \text{Zinc Content (g/l)}$$

Example:

$$\frac{0.8 \text{ mg/l AAS}}{1000} \times 10000 = 8.0 \text{ g/l Zn}$$

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.
- ** Volumes given exact to a decimal point must be measured with a volumetric pipette.

Determination of Zn and Ni concentration by XRF

Manufacturer's method · Version 03 · Doc.-No. A0001526-03

Principle:
XRF in "concentration mode".
Calibration with standard **sample**s with known concentrations.

Reagents required*:

- (DI) water
- Zn/Ni baths of different concentrations (certified materials) for calibration.
- **Example** Zinni AC AF 210 (acid Zn/Ni):
  - c(Zn/Ni) = 10/10g/l, 20/20g/l, 30/30g/l
- **Example** Zinni AL 450 (alkaline Zn/Ni):
  - c(Zn/Ni) = 5/0.5g/l, 10/1.0g/l, 15/1.5g/l
- **Important: All Standards have to be made up containing the same sample matrix to achieve proper results**.

Please follow the general regulations for hazardous materials in the Material safety data sheets!
The analytical procedure is to be followed exactly.
Be sure to observe all local regulations concerning the permitted limits for pollutants.

Equipment required:

- XRF system (must be able to separate Zn Ni lines, e.g. FISCHER XDAL)
- Measuring cell (for Zn/Ni-solution)
- (transparent) foil (to cover measuring cell)

Equipment adjustment:

Electrolyte volume	few ml (depends on measuring cell volume)
Electrolyte temperature	Room temperature (22 °C)

XRF parameter:

Tube voltage	e.g. 50 kV
Collimator	e.g. 0.2 mm x 0.2 mm
Measuring time	e.g. 60 s
Primary filter	Ni
Drift compensation	On

Procedure:

Calibration of application:

- Generate an application for the measurement of solutions following the instructions of your systems manual.
- Use calibration standards which cover the (expected) concentration range of the solutions to be measured. For this fill some amount of the calibration standard solution into the measuring cell (see step "**Sample**").

Sample:

- The surface of the solution to be analyzed must be flat. The following steps describe a method to achieve a flat solution surface:
- Fill the measuring cell with the solution. The liquid surface must show a convex curvature.
- Cut out a quadratic piece of foil to cover the measuring cell.
- Cover the measuring cell with the foil and clamp the foil (e.g. by a clamp ring) that the foil surface is flat. No foil break and no air bubbles may occur.

Measurement:

- Place the prepared measurement cell in the measurement chamber.
- Focus on the surface of the foil.
- Start the measurement(s).

Evaluation:

The **evaluation** is carried out automatically by the XRF software.

Statistic:

Repeatability: RSD (n=6) < 3%

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.

Nickel and Zinc by ICP-OES

Manufacturer's method · Version 04 · Doc.-No. A0001581-04

Principle: Inductively Coupled Plasma - Optical Emission Spectrometry

Inductively coupled plasma optical emission spectrometry.
The aqueous **sample** solution is brought into a fine aerosol with the aid of a nebulizer and an Argon carrier gas and is subsequently led into an inductively coupled Argon plasma. The radiation emitted from the analyte elements present in the plasma is recorded wavelength-resolved by a spectrometer what allows for their identification. The ICP-OES instrument is calibrated by the analysis of aqueous standard solutions. Yttrium is used as internal standard.

Reagents required:

- **Nickel** stock solution, c = 1000 mg/l
- **Zinc** stock solution, c = 1000 mg/l
- Yttrium stock solution, c = 1000 mg/l
- Nitric acid (HNO_3, 65%, suprapur, ρ=1.40 g/ml)
- purified water of analytical grade

Please follow the general regulations for hazardous materials in the Material safety data sheets!
The analytical procedure is to be followed exactly.
Be sure to observe all local regulations concerning the permitted limits for pollutants.

Equipment required:

- Pipettes
- 50 ml graduated flask
- ICP-OES,

Operating conditions:
ICP-OES, Type Varian ICP 720ES
Software: ICP Expert II software

Analyte wavelengths	Ni 216.555 nm, Zn 202.548 nm

Spectrometer

Replicates		Replicate read time	
5		30 s	

Plasma

Power		Plasma gas flow	
1200 W		16.5 l/min	

Nebulizer pressure		Auxiliary gas flow	
200 kPa		1.5 l/min	

Peristaltic Pump Program / Autosampler

Pump rate		Instr. stabil. delay	
20 rpm		30 s	

Sample uptake delay		Fast pump	
30 s		on	

Rinse time		-	
30 s		-	

Max. rinse time		Smart rinse	
-		off	

Calibration

Calibration mode		Weighted Fit	
External calibration		on	

Curve Type		Thru Blank	
Linear		off	

Sequence Editor

Begin with calibration		Include a blank in c.	
on		on	

Nebulizer	Seaspray® (concentric, Meinhardt-type)
Spraychamber	Twister (Cyclone)

When using other instruments the parameters must be adjusted correspondingly!

For calibration, the standards listed below have to be prepared.

Standard	Ni (mg/l)	Zn (mg/l)
1	0	0
2	1	10
3	2	20
4	5	50
5	10	100

ICP-OES, Type Spectro Spectroblue
Software: Smart Analyzer Vision

Analyte wavelengths	Zn 213.856 nm, Ni 231.604 nm

Spectrometer

Replicates		Measurement time	
3		36 s (standard)	

Plasma

Power		Plasma gas flow	
1200 W		16.5 l/min	

Nebulizer gas flow		Auxiliary gas flow	
0.8 l/min		0.8 l/min	

Peristaltic Pump Program / Autosampler

Pump rate (during measurements)		Overall rinse sample	
30 rpm		60 s/60 rpm	

Pre-rinse sample		Rinse autosampler	
30 s/60 rpm		30 s/30 rpm	

Ring pos.		Method	
Rinse		Rinse	

Calibration

Application type		Regression	
normal		Calc. f(x)	

Nebulizer	Burgener T 2002 Nebulizer
Spraychamber	Twister (Cyclone)

When using other instruments the parameters must be adjusted correspondingly!

The following standards have to be prepared by diluting the stock solutions with 1 % HNO_3:

	Y	Zn	Ni
Blank	1 mg/l	0	0
Standard 1	1 mg/l	1 mg/l	1 mg/l
Standard 2	1 mg/l	2 mg/l	2 mg/l
Standard 3	1 mg/l	5 mg/l	5 mg/l
Standard 4	1 mg/l	10 mg/l	10 mg/l
QC standard	1 mg/l	5 mg/l	5 mg/l

Procedure:

Sample Preparation

The **sample**s have to be diluted by a factor of 1:200 to 1:10000 with 1 % HNO_3 and the concentration of Y has to be adjusted to 1 mg/l.

The calibration of the ICP–OES instrument is done by external calibration under the use of Yttrium as internal standard:

The calibration standards are prepared from stock solutions (1000 mg/l) by diluting them with 1% HNO_3. For the determinations the Y 371.029 nm emission line was used as internal standard. The measuring sequence starts with the blank **sample** and continues with standard and sample solutions. For the verification of the calibration a control sample has to be measured after the calibration and as last sample. The measured intensity for the sample has to be within the calibration range, otherwise another dilution has to be chosen.

Evaluation:

The concentration of the **sample** is determined under the use of external calibration and an internal standard (Yttrium (Y)) with the aid of the Varian ICP Expert II software.

Formula for **calculation**:

Conc. Ni/Zn (diluted **sample**) • dilution factor = conc. Ni/Zn (undiluted sample)

Example:
5 mg/l Ni •·200 = 1 g/l Ni

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.
- ** Volumes given exact to a decimal point must be measured with a volumetric pipette.

Determination of Sodium hydroxide by Titration

Manufacturer's method · Version 03 · Doc.-No. EXT-0314-TIT-03

Required reagents*

- Sodium cyanide p.A., e. g. Messrs. Merck
- Indicator solution (0.2 % 4-(4-Nitrophenylazo)resorcinol (**CAS Nr. 74-39-5**) in 80 % isopropanol), Aldrich
- Hydrochloric volumetric solution 0.5 M, e.g. Messrs. Kraft
- DI water

Preparation of the standard

- 1.0 g NaCN are placed into a 100 ml beaker and dissolved with
- 50 ml DI water. Now add
- 5 drops indicator solution (colour standard).

Be sure to observe all local regulations concerning the limiting values of pollutants including the general regulations on hazardous materials!

Procedure

- 2.0 ml** **sample** is pipetted into a 100 ml beaker and mixed with
- 1.0 g NaCN. After addition of
- 5 drops of indicator solution and
- 50.0 ml DI water titrate with
- 0.5 M HCl volumetric solution until the colour of the solution turns into the same as the standard.

Calculation

Consumption (ml/l)HCl(0.5M) x 10*** = g/l NaOH

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.
- ** Volumes given exact to a decimal point must be measured with a volumetric pipette.
- *** For the purposes of **calculation**, the factor of the volumetric solutions is assumed to be 1.000 and is therefore not taken into account in the **example**s.

Determination of Sodium Carbonate by Titration

Manufacturer's method · Version 10 · Doc.-No. EXT-0526-TIT-10

Principle: Acid base titration

Reagents and Equipment required*:

- Hydrochloric acid volumetric solution 0.5 M (0.5 N)
- Barium chloride 20 %
- Phenolphthalein indicator solution
- Burette

Please follow the general regulations for hazardous materials in the Material safety data sheets!
The analytical procedure is to be followed exactly.
Be sure to observe all local regulations concerning the permitted limits for pollutants.

Procedure:

Prepare a first sample as follows:

- Place 2.5 ml** of the **sample** into an Erlenmeyer flask and mix with approx.
- 80 ml DI water. To this solution
- **exactly 100 µL** phenolphthalein indicator solution is added. Now titrate with
- 0.5 M hydrochloric acid volumetric solution until the solution is decolorized. (**Consumption 1**).

Prepare a second sample as follows:

- Place 2.5 ml** **sample** into a Erlenmeyer flask and mix with approx.
- 80 ml DI water. To this solution
- 20 ml barium chloride solution and
- **exactly 100 µL** phenolphthalein indicator solution is added. Now titrate with
- 0.5 M hydrochloric acid volumetric solution until the color changes from pink to colorless or until no change in color can be noticed (**sample** solution can keep a slight pink color) (Consumption 2).
- The indicator should stay colorless for at least 10 seconds. Another change in color of the indicator doesn't have to be taken into consideration.

Alternative: The Titration can be conducted as a setpoint titration using a titroprocessor equipped with a calibrated pH electrode to determine the endpoint at pH 8.5.

In case of lower amounts of Sodium Carbonate or irritating results the initial volume can be increase to 5.0 ml. The **calculation** factor has to be adapted to 10.6.

The detection limit of measurement is 10 g/l Na_2CO_3. Do not hand out lower contents.

Evaluation

Content of sodium carbonate

(**Consumption 1** - Consumption 2) × 21.2*** = g/l Na_2CO_3

Example:
Consumption 1: 19.26 ml 0.5 M Hydrochloric acid volumetric solution
Consumption 2: 16.83 ml 0.5 M Hydrochloric acid volumetric solution

Calculation:

Content of sodium carbonate

- (19.26 ml – 16.83 ml) × 21.2 = 51.5 g/l Na_2CO_3

Quality assurance: For maintenance, calibration of measuring devices and the use of standards and reference materials please refer to the corresponding guidelines.

- * Unless otherwise stated, these are always analytical grade substances.
- ** Volumes given exact to a decimal point must be measured with a volumetric pipette.
- *** For the purpose of **calculation**, the factor of the standard solution is assumed to be 1.000 and is therefore not taken into account in the **example**s.

Literal transcription of the manufacturer's original document (Atotech Deutschland GmbH · Doc. 1112 · v18). Content verified automatically against the original — text, values, formulas and highlighted warnings — on 03.09.2026. Approved on 03.09.2026.

Document reproduced by REYM from the technical data sheet of Atotech Deutschland GmbH, document 1112 version 18 dated 25.11.2015, preserving the original text. The technical content belongs to the manufacturer.

Manufacturer's notice: This sheet shall serve for your information. The content of the sheet is based on our best knowledge. We do not undertake any duty to advise. The contents of the sheet shall not be considered as an agreement on condition and quality of the goods delivered. The decision to use our products for its production purposes is the responsibility of our customer. We can accept liability only for the quality of our products at delivery. If difficulties arise in the application of our products, we propose that you contact our technical service department.

P.O. Box: 21 07 80 10507 Berlin, Germany Erasmusstraße 20 10553 Berlin, Germany · phone +49 (0) 30 349 85 0 fax +49 (0) 30 349 85 777