



CERTIFICATE OF ANALYSIS

ERM®-AE140

Pd, enriched in ¹⁰⁶ Pd, in 20 % hydrochloric acid					
Certified quantity	Unit	Certified value 1)	Uncertainty 2)		
Mass fraction w(106Pd)	mg/kg	20.24	0.05		
Isotope amount ratio $n(^{102}Pd)/n(^{106}Pd)$	-	0.0000791	0.0000011		
Isotope amount ratio $n(^{104}Pd)/n(^{106}Pd)$		0.001247	0.000011		
Isotope amount ratio $n(^{105}Pd)/n(^{106}Pd)$	- 1	0.007518	0.000030		
Isotope amount ratio $n(^{108}Pd)/n(^{106}Pd)$	-	0.004785	0.000022		
Isotope amount ratio $n(^{110}Pd)/n(^{106}Pd)$	-	0.001156	0.000011		

¹⁾ The certified values of this European Reference Material (ERM) are traceable to the International System of units (SI) in the shortest possible way, by calibrating all instruments (balance, mass spectrometer) against SI-traceable calibrators. Measurements calibrated against this ERM will, therefore, also be traceable to the SI.

This certificate is valid for 10 years for units with unbroken seal stored under required conditions. This validity may be extended as further evidence of stability becomes available.

NOTE

European Reference Material ERM®-AE140 was produced and certified under the responsibility of BAM Bundesanstalt für Materialforschung und -prüfung according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Berlin, Germany, January 2015

BAM Department 1 Analytical Chemistry; Reference Materials BAM Division 1.1 Inorganic Trace Analysis

Prof. Dr. U. Panne (Head of Department)

Dr. N. Jakubowski (Head of Division)

²⁾ The uncertainty of the certified value is the expanded uncertainty U with a coverage factor of k = 2 corresponding to a level of confidence of about 95 % estimated in accordance with international guidelines such as JCGM 100:2008 and EURACHEM/CITAC 2012; this includes the repeatability of the measurement and of the determination of correction factors for systematic deviations. Standard uncertainties can be obtained from the certification report.

Indicative Values					
Quantity	Unit	Value 1)	Uncertainty 2)		
Mass fraction w(Pd)	mg/kg	20.54	0.05		
Isotope amount fraction n(102Pd)/n(Pd)	-	0.0000780	0.0000010		
Isotope amount fraction n(104Pd)/n(Pd)	-	0.001229	0.000010		
Isotope amount fraction n(105Pd)/n(Pd)	-	0.007408	0.000030		
Isotope amount fraction n(106Pd)/n(Pd)	-	0.98543	0.00005		
Isotope amount fraction n(108Pd)/n(Pd)	-	0.004716	0.000022		
Isotope amount fraction n(110Pd)/n(Pd)	-	0.001139	0.000011		
Molar mass of Pd in solution M(Pd)	g/mol	105.90731	0.00008		

¹⁾ The atomic masses used for the indicative values were taken from De Laeter et al. 2003.

When Pd in the **sample shows no isotope variation** against the IUPAC values the measurement uncertainty can be reduced by applying a quasi double IDMS analysis. The isotopic composition of Pd in the used back-spikes and the isotopic composition of Pd in the sample are equal and cancel when the equations of IDMS and reverse IDMS are combined to the double IDMS equation. To offer the analyst this quasi double IDMS approach, the Pd mass fractions in ERM-AE140 was calculated with treating the IUPAC values for the molar mass in the back-spike and the isotopic abundance of Pd-106 in the back-spike as constants. The so calculated mass fractions can be used for Pd quantification by IDMS, when the same IUPAC values were used as constants. It has to be stressed here, that this quasi double IDMS approach only is valid, when **no isotopic variation** of the Pd in the sample occurs.

Derived quantity values for quasi-double IDMS. The expanded uncertainties (k = 2) were calculated with IUPAC values treated as constant: The isotope amount fraction $n(^{106}Pd)/n(Pd) = 0.2733$ (Berglund and Wieser 2011) and the atomic weight $A_r(Pd) = 106.42$ (Wieser et al. 2013) have been used.

Additional Material Information					
Quantity	Unit	Value	Uncertainty		
Mass fraction w(106Pd)	mg/kg	20.235	0.011		
Mass fraction w(Pd)	mg/kg	20.535	0.011		

DESCRIPTION OF THE SAMPLE

The spike isotopic reference material ERM-AE140 is a solution of isotopically enriched Pd in 20 % hydrochloric acid and filled in flame-sealed quartz ampoules containing approximately 7 mL solution. This material is designed to serve as isotopically enriched analogue or so-called spike in Isotope Dilution Mass Spectrometry (IDMS) for the quantification of Pd.

ANALYTICAL METHOD USED FOR CERTIFICATION

The certified values were determined by reverse IDMS using multi-collector ICPMS. The measurements were calibrated using high purity Pd for the preparation of the back-spike. More details can be found in the certification report, which can be requested from BAM.

PARTICIPANTS

BAM Division 1.1

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²⁾ The uncertainties of the indicative values are the expanded uncertainties U with a coverage factor of k=2 corresponding to a level of confidence of about 95 % estimated in accordance with international guidelines such as JCGM 100:2008 and EURACHEM/CITAC 2012.

INSTRUCTIONS FOR USE

The ampoule should be shaken for some minutes before opening in order to combine the solution with potential water condensation in the neck, which may occur when stored upright for a longer time. Transfer of the solution to another container preferentially should be done by pouring. Contamination may occur when pipettes or other liquid handling devices are used for this purpose. Therefore it is recommended to use only pre-cleaned devices or labware. Contamination could lead to a change in the isotope composition.

When an ampoule is to be opened, the whole ampoule should be carefully wiped with a clean, damp cloth and the body of the ampoule should be wrapped in absorbent material (e.g. clean cloth). Then an ampoule file with a diamond or WIDIA blade is used to score the neck of the ampoule with a quarter to a half circle. Holding the ampoule steady and with both hands, medium thumb pressure should be applied with both thumbs to the stem to snap it. Correctly done, the stem should break where scored.

SAFETY INFORMATION

The usual laboratory safety precautions apply.

ERM-AE140 is an acidic solution sealed in quartz ampoules, which contains 20 % hydrochloric acid. All appropriate safety precautions, including the use of gloves and safety glasses, should be taken.

STORAGE

This CRM should be stored under normal laboratory conditions (between 5 °C and 25 °C) at places, where the risk of mechanical damage is low.

BAM cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

REFERENCES

Berglund, M. and Wieser, M. E., 2011. Isotopic compositions of the elements 2009 (IUPAC Technical Report). Pure Appl. Chem. 83, 397-410.

De Laeter, J. R., Bohlke, J. K., De Bievre, P., Hidaka, H., Peiser, H. S., Rosman, K. J. R., and Taylor, P. D. P., 2003. Atomic weights of the elements: Review 2000 - (IUPAC technical report). Pure Appl. Chem. 75, 683-800.

Wieser, M. E., Holden, N., Coplen, T. B., Böhlke, J. K., Berglund, M., Brand, W. A., De Bièvre, P., Gröning, M., Loss, R. D., Meija, J., Hirata, T., Prohaska, T., Schoenberg, R., O'Connor, G., Walczyk, T., Yoneda, S., and Zhu, X. K., 2013. Atomic weights of the elements 2011 (IUPAC Technical Report). Pure Appl. Chem. 85, 1047-1078.

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NOTE

A detailed technical report describing the production, characterisation as well as the analytical procedures applied and the treatment of the analytical data used to certify ERM®-AE140 is available on request from BAM (http://www.rm-certificates.bam.de/de/certificates/isotopic materials/index.htm).

Supply of Reference Materials by: BAM Bundesanstalt für Materialforschung und -prüfung

Richard-Willstätter-Str. 11, D-12489 Berlin, Germany Phone: +49 30 8104 2061 – Fax: +49 30 8104 1117 e-mail: sales.crm@bam.de – internet: www.bam.de

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SAFETY DATA SHEET FOR

according to Regulation (EC) No 1907/2006

"ERM-AE140"

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Section 1: Identification of the substance/mixture and of the company/undertaking

1.1. Product identifier

Product name: ERM-AE140

Product information: Palladium aqueous solution in 20 % hydrochloric acid with an approximate

Pd mass fraction of 20 µg/g and a total volume of 7 mL in flame-sealed

quartz ampoules

1.2. Relevant identified uses of the substance or mixture and uses advised against

Certified Reference Material to be used for spiking samples in isotope dilution mass spectrometry for the quantification of Pd mass fractions. Any other use is discouraged.

1.3. Details of the supplier of the safety data sheet

Supplier / Producer: Bundesanstalt für Materialforschung und -prüfung (BAM)

Unter den Eichen 87, 12205 Berlin, Germany

Phone: +49 (0)30 8104-0 Fax: +49 (0)30 8104-7-2222 Homepage: <u>www.bam.de</u> E-Mail: info@bam.de

Contact person: Dr. Jochen Vogl, e-mail: jochen.vogl@bam.de
Issuing person: Dr. Jochen Vogl, e-mail: jochen.vogl@bam.de

1.4. Emergency telephone number

Emergency telephone: +49 (0)30 30686700

Giftnotruf Berlin

Charité-Universitätsmedizin Berlin Campus Benjamin Franklin Hindenburgdamm 30

12203 Berlin

To avoid language problems and in case of nonavailability it is recommended to contact your national poison control centre. A list of national poison control centres inside the EU can be obtained at:

http://ec.europa.eu/growth/sectors/chemicals/poison-centres/index_en.htm

For poison centres outside the EU the information is listed at the world

directory of poison control centres at the WHO homepage:

http://www.who.int/gho/phe/chemical_safety/poisons_centres/en/

2. Hazards identification

2.1. Classification of the substance or mixture

Classification (Regulation (EC) No 1272/2008)

Skin Irritation, Category 2 H315: Causes skin irritation

Eye Irritation, Category 2 H319: Causes serious eye irritation
STOT SE 3 H335: May cause respiratory irritation

Corrosive to metals, Category 1 H290: May be corrosive to metals

Classification (67/548/EEC or 1999/45/EC)

Xi R36: Irritating to eyes



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R37: Irritating to respiratory system

R38: Irritating to skin

Cut-off value for palladium(II) chloride and palladium compounds is the generic cut-off value of 0.1 %, as no other value is stated in Annex VI of council regulation EC 1272/2008. Components of mixtures with mass fractions below the cut-off value need not being considered for classification. The mass fraction of palladium(II) chloride is < 0.005 % and thus below the cut-off value. The classification therefore derives from hydrochloric acid with c = 20 %.

2.2. Label elements

Labelling according to Regulation (EC) No 1272/2008:

Hazard pictogram:





Signal word:

Danger

Hazard statements:

H315 Causes skin irritation.

H319 Causes serious eye irritation
H335 May cause respiratory irritation
H290 May be corrosive to metals

Precautionary statements:

P261 Avoid breathing dust/fume/ gas/mist/vapours/spray.

P280 Wear protective gloves/protective clothing/eye protection/face protection.

P301 + P330 + P331 IF SWALLOWED: Rinse mouth. Do NOT induce vomitting

P304 + P340 IF INHALED: Remove victim to fresh air and keep art rest in a position

comfortable for breathing.

P305 + P351 + P338 IF IN EYES: Rinse cautiously with water for several minutes. Remove

contact lenses, if present and easy to do. Continue rinsing.

P312 Call a POISON CENTER/ doctor if you feel unwell

Restricted to professional users.

For the full text of the H-Statements as well as S- and R-phrases mentioned in this Section, see Section 16.

Section 3: Composition/information on ingredients

3.1. Substances

Does not apply. Product is prepared as mixture from substances listed under section 3.2.

3.2. Mixtures

Chemical nature: Palladium chloride in hydrochloric acid solution.



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Hazardous components (Regulation (EC) No 1272/2008)

Chemical Name (Concentration)

CAS-No. EC-No./Registration Index-No. Classification of the pure component

number

Hydrochloric acid (>= 10 % -=<25 %)

7647-01-0 231-595-7 017-002-01-X Skin irritation 2, H315

Eye irritation 2, H319 STOT SE 3, H335

Corrosive to metals, Category 1, H290

Palladium(II) chloride (classification of pure substance)

7647-10-1 231-596-2 Skin irritation, Category 2, H315

Skin sensitization, Category 1, H317 Eye irritation, Category 2, H319

Cut-off value for palladium(II) chloride is the generic cut-off value of 0.1 %, as no other value is stated in Annex VI of council regulation EC 1272/2008. Components of mixtures with mass fractions below the cut-off value need not being considered for classification.

Hazardous components (67/548/EEC)

Chemical Name (Concentration)

CAS-No. EC-No./Registration Index-No. Classification of the pure component

Number

Hydrochloric acid (>= 10 % -=<25 %)

7647-01-0 231-595-7 017-002-01-X Xi, R36/37/38

Palladium(II) chloride (classification of pure substance)

7647-10-1 231-596-2 Xi, irritant, R36/38

R43

Cut-off value for palladium(II) chloride is the generic cut-off value of 0.1 %, as no other value is stated in Annex VI of council regulation EC 1272/2008. Components of mixtures with mass fractions below the cut-off value need not being considered for classification.

For the full text of the R-phrases mentioned in this Section, see Section 16.

Section 4: First aid measures

4.1. Description of first aid measures

After inhalation: fresh air. Consult doctor in case of complaints.

After skin contact: wash off with plenty of water. Remove contaminated clothing.

After eye contact: rinse out with plenty of water. Call in ophthalmologist.

After swallowing: immediately make victim drink water (two glasses at the most). Consult a physician.

4.2. Most important symptoms and effects, both acute and delayed

Irritation and corrosion, Cough, Headache, Stomach ache Risk of severe eye damage, risk of breathing dysfunction

4.3. Indication of any immediate medical attention and special treatment needed

No information available



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Section 5: Fire-fighting measures

5.1. Extinguishing media

Use extinguishing measures that are appropriate to local circumstances and the surrounding environment. For this substance / mixture no restrictions on extinguishing media are known.

5.2. Special hazards arising from the substance or mixture

Not combustible.

Ambient fire may liberate hazardous vapours (hydrogen chloride gas).

5.3. Advice for fire fighters

Do not stay in dangerous zone without self-contained breathing apparatus. In order to avoid contact with skin, keep a safety distance and wear suitable protective clothing.

Further information

Suppress gases/vapours with a water spray jet. Prevent fire extinguishing water from contamination surface water or the ground water system.

Section 6: Accidental release measures

6.1. Personal precautions, protective equipment and emergence procedures

Do not breathe vapours, aerosols. Avoid substance contact. Ensure adequate ventilation.

Wear protective glasses and gloves. See section 8

6.2. Environmental precautions

Do not empty into drains.

6.3. Methods and materials for containment and cleaning up

Take up with liquid-absorbent and neutralizing material. Forward for disposal. Clean up affected area.

6.4. Reference to other sections

Information on disposal see section 13.

Section 7: Handling and storage

7.1. Precautions for safe handling

Work under hood. Do not inhale substance. Avoid generation of vapours/aerosols. Observe label precautions. Wear protective equipment, see section 8

Keep general hygiene standards for laboratories.

7.2. Conditions for safe storage, including any incompatibilities

Not use metal containers. Keep container tightly closed. Keep in a well-ventilated place.

7.3. Specific end use

Not applicable



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Section 8: Exposure controls/personal protection

8.1. Control parameters

Components with workplace parameters

Components

Basis Value Threshold limits Remarks

Hydrochloric acid (7647-01-0)

Directive 2000/39/EC Short Term Exposure

Limit (STEL): 15 mg/m³ (10 ppm) 15 minutes

Occupational exposure

limit value 8h 8 mg/m³ (5 ppm) 8 h average

Palladium(II) chloride (7647-10-1)

No such values available

Recommended monitoring procedures

Methods for measurement of the workplace atmosphere have to correspond to the requirements of standards DIN EN 482 and DIN EN 689.

8.2. Exposure controls

Personal protective equipment

Protective clothing should be selected specifically for the working place, depending on concentration and quantity of the hazardous substances handled. The resistance of the protective clothing to chemicals should be ascertained with the respective supplier.

Hand protection:

Full contact: Glove material: Latex or polyvinylchloride; nitrile rubber

Glove thickness: 0. 5 mm; 0.11 mm

Break through time: > 8 h

Splash contact: Glove material: Latex or polyvinylchloride; nitrile rubber

Glove thickness: 0.5 mm; 0.11 mm

Break through time: > 8 h

The protective gloves to be used must comply with the specifications of EC Directive 89/686/EEC and the resultant standard EN374.

This recommendation applies only to the product stated in the safety data sheet and supplied by us as well as to the purpose specified by us. When dissolving in or mixing with other substances and under conditions deviating from those stated in EN374 please contact the supplier of CE-approved gloves.

Eye protection:

Safety glasses

Hygiene measures:

Immediately change contaminated clothing. Apply skin-protective barrier cream. Wash hands and face after working with substance.

Section 9: Physical and chemical properties

9.1. Information on basic physical and chemical properties

Appearance colourless liquid
Odour slightly pungent

Odour threshold no information available



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pH < 0 at 20 °C, for the hydrochloric acid component,

literature data

Melting point/freezing point between 0 °C and -30 °C

Initial boiling point and boiling range > 80°C

Flash point not required, inorganic substance

Evaporation rate no data available
Flammability not combustible
Upper/lower flammability or explosive limits no data available
Vapour pressure no data available
Vapour density no data available

Relative density ca. 1.098 g/cm³ at 20 °C, based on tabulated data for

Solubility(ies) water soluble
Partition coefficient: n-octanol/water no data available
Auto-ignition temperature no data available
Decomposition temperature no data available
Viscosity no data available

Explosive properties not classified as explosive

Oxidizing properties not oxidizing

9.2. Other information

No other information available.

Section 10: Stability and reactivity

10.1. Reactivity

See section 10.3.

10.2. Chemical stability

Stable under normal storage conditions (0 – 40 °C)

10.3. Possibility of hazardous reactions

Generates dangerous gases or fumes in contact with:

Metals

Violent reactions possible with generally known reaction partners of water.

10.4. Conditions to avoid

No information available

10.5. Incompatible materials

Increased reactivity with:

Metals, metal alloys, Alkali metals, Alkaline earth metals, Ammonia, alkalines, acids

Unsuitable working materials:

Metals, metal alloys

10.6. Hazardous decomposition products

no information available



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Section 11: Toxicological information

11.1. Information on toxicological effects

Acute oral toxicity:

Symptoms: Irritations of mucous membranes in the mouth, pharynx, oesophagus and gastrointestinal tract.

Acute inhalation toxicity

Symptoms: mucosal irritations, cough, shortness of breath, possible damage of respiratory tract.

Acute dermal toxicity

This information is not available.

Skin irritation:

Causes skin irritation.

Serious eye damage/irritation:

Causes serious eye irritation.

Respiratory or skin sensitization:

Sensitization possible in predisposed persons.

Carcinogenicity

This information is not available.

Germ cell mutagenicity

This information is not available.

Reproductive toxicity

This information is not available.

Specific target organ toxicity - single exposure

This information is not available.

Specific target organ toxicity - repeated exposure

This information is not available.

Aspiration hazard

This information is not available.

11.2. Further information

CMR effects:

Carcinogenicity:

This information is not available.

Quantitative data on the toxicity of this product are not available.

Handle in accordance with good industrial hygiene and safety practice.

Section 12: Ecological information

12.1. Toxicity

No information available.

12.2. Persistence and degradability

No information available.

12.3. Bio accumulative potential

No information available.



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12.4. Mobility in soil

No information available.

12.5. Results of PBT and vPvB assessment

No information available.

12.6. Other adverse effects

No information available.

Further information on ecology

Do not allow to enter waters, waste water, or soil!

Section 13: Disposal considerations

13.1. Waste treatment methods

Product

Chemicals must be disposed of in compliance with the Directive on waste 2008/98/EC as well as other respective national regulations. Handle uncleaned containers like the product itself.

Packaging

The product packaging must be disposed of in compliance with the country-specific regulations or must be passed to a packaging return system.

Section 14: Transport information

Land transport (ADR/RID)

14.1. UN number 1789

14.2. UN proper shipping name HYDROCHLORIC ACID

Inland waterway transport (ADN)

Not relevant

Air transport (IATA/ICAO)

14.1. UN number 1789

14.2. UN proper shipping name HYDROCHLORIC ACID

14.3. Transport hazard class(es)
14.4. Packing group
14.5 Environmental hazards
14.6. Special precautions for users

Sea transport (IMDG)

14.1. UN number 1789

14.2. UN proper shipping name HYDROCHLORIC ACID



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14.3. Transport hazard class(es)
14.4. Packing group
14.5 Environmental hazards
14.6. Special precautions for users

EmS F-A S-B

14.7. Transport in bulk according to Annex II of Marpol 73/78 and the IBC code

Not relevant

Additional Transport Information:

Product ERM-AE140 fulfils the limits for excepted quantities according to ADR and IMDG:

ADR: Limited quantity (LQ): 1 L

Excepted quantity (EQ) Code: E2 maximum net quantity per inner packaging: 30 mL

maximum net quantity per outer packaging: 500 mL

Transport category 2
Tunnel restriction code E
IMDG Limited quantities (LQ): 1L

Excepted quantities (EQ): E2 maximum net quantity per inner packaging: 30 ml

Maximum net quantity per outer packaging: 500 ml

The transport regulations are cited according to international regulations and in the form applicable in Germany. Possible national deviations in other countries are not considered.

Section 15: Regulatory information

15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture

EU regulations

Major Accident Hazard 96/82/EC

Legislation Directive 96/82/EC does not apply

Occupational restrictions Take note of Directive 94/33/EC on the protection of young people at

work

National legislation

Storage class (TRGS 510): 8B

15.2 Chemical safety assessment

For this product a chemical safety assessment is not required and therefore was not carried out.

Section 16: Other information

Full text of H-Statements referred to under sections 2 and 3

H290 May be corrosive to metals

H315 Causes skin irritation

H317 May cause an allergic skin reaction
H319 Causes serious eye irritation

H335 May cause respiratory irritation



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Full text of R-phrases referred to under sections 2 and 3 R36/38 Irritating to eyes and skin

R36/37/38 Irritating to eyes, respiratory system and skin R43 May cause sensitisation by skin contact

Full text of S-phrases referred to under sections 2 and 3

S26 In case of contact with eyes, rinse immediately with plenty of water and

seek medical advice.

S45 In case of accident or if you feel unwell seek medical advice immediately.

Release management: Regulation (EC) No 453/2010

The information contained herein is based on data considered to be accurate and on the present state of our knowledge. It characterizes the sample with regard to the appropriate safety precautions. However, no warranty is expressed or implied regarding the accuracy of these data or the results to be obtained from the use thereof.