

Recycled gypsum **(RC-gypsum)**

Initial test for recycling plants, quality management, quality requirements and analysis methods

Bundesverband der Gipsindustrie e.V.

December 2013

Initial test for recycling plants quality management

Initial test of all quality parameters before delivery and QM audit

The quality management audit before the first delivery is performed to examine the conditions of acceptance, storage, processing of waste depending on their origin (H1, H2), the discharge and disposal of impurities, the correct conducting of the analyses complying with standard processes and in certified laboratories and the legally required questions (acceptance permit, BImSchG-permit, product manufacturing).

Requirements are the conditions for acceptance separating origin H1 without adhesion = prefabricated houses, further processing, scrap new building and H2 deconstruction, demolition.

The adherence to the specifications in the following chart part 1 (technical parameters) has to be proven by batch. The adherence to the quality requirements of the following chart part 2 (health parameters) and 3 (eluate parameters) has to be proven at least once a year (only H1-acceptance) or four times per year. For mobile plants these parameters must additionally be proven after putting-into-operation following a change of location (initial test).

The regulations on the adherence to the quality requirements as well as the determination of sample taking rhythms and analyses are an essential part of the delivery and taking delivery contracts for RC gypsum to be concluded.

Quality requirements

Quality parameters		Target value	Remark
Part 1 technical parameter			
Particle size [mm]		≤ 1	Plant specific upward deviations permitted
Humidity [% w/w]	H ₂ O	≤ 5	< 10% w/w (when agreed particle size > 1mm (as bulk material))
Calcium sulfate dihydrate [% w/w]	CaSO ₄ x 2H ₂ O	≥ 85	Plant specific deviations down to 80 % w/w min. only after special agreement permitted
Org. carbon [% w/w]	TOC	≤ 1,0	Plant specific deviations up to 1,5 % w/w max. only after special agreement
Elimination Visible impurities		visual assessment	Residues of plasterboard lamination or wall board lamination also count as impurities
Odour		neutral	
Magnesium salts water soluble [% w/w]	MgO	< 0,02	Plant specific deviations up to 0,1 % w/w MgO max. only after special agreement ¹⁾

Quality parameters		Target value	Remark
Natrium salts water soluble [% w/w]	Na ₂ O	< 0,02	Plant specific deviations up to 0,04 % w/w Na ₂ O max. only after special agreement ¹⁾
Potassium salts water soluble [% w/w]	K ₂ O	< 0,02	Plant specific deviations up to 0,06 % w/w max. only after special agreement permitted ¹⁾
Chloride [% w/w]	Cl ⁻	< 0,01	Plant specific deviations up to 0,02 % w/w only after special agreement permitted ¹⁾
pH value		5 - 9	
1) Observe interaction Cl with Na and Mg.			
Part 2 Health parameters			
Fluoride [% w/w]	F ⁻	< 0,02	
Radioactivity acc. to RP112 [index]		< 0,5	
Material must be asbestos-free			

Quality parameters		Target value	Remark
Trace element contents acc. to maximum values „Beckert Study“ [mg/kg]	As	< 4	Values can be adjusted to new human-toxicological assessments and limits
	Sb	---	
	Be	< 0,7	
	Pb	< 22	
	Cd	< 0,5	
	Cr	< 25	
	Co	<4	
	Cu	< 14	
	Mn	< 200	
	Mo	---	
	Ni	< 13	
	Hg	< 1,3	
	Se	< 16	
	Te	< 0,3	
	Tl	< 0,4	
	V	< 26	
	Zn	< 50	
PAK (EPA)	< 0,2		
Sulphur (primary)	S		Odour test

Analysis methods

Note:

In order to determine the RC gypsum quality parameters further analysis methods can be used as well provided that the results are consistent with the results determined by the reference techniques.

Relevant parameters and corresponding analysis methods are defined in the delivery and taking delivery contracts for RC gypsum. When changing the legal basic conditions the mentioned analysis methods can be adjusted to new requirements.

The following methods were discussed with the ad-hoc working group “analysis of recycled gypsum” of the *Wissenschaftlichen Beirates der Forschungsvereinigung der Gipsindustrie e.V.* (scientific advisory council of the research association of the German gypsum industry) in cooperation with the company Dorfner Anzaplan GmbH.

Participants: Dr. Thomas Bach (Dorfner Anzaplan GmbH), Helmut Günther (Hilliges Gipswerk GmbH), Prof. Dr. Hans-Ulrich Hummel (Knauf Gips KG), Dr. Hans-Jörg Kersten (Bundesverband der Gipsindustrie e.V.), Dr. Hans-Ulrich Kothe (Casea GmbH), Gundolf Krüger (Knauf Gips KG), Elmar Limley (Siniat GmbH), Sören Olejnik (VG Orth GmbH & Co. KG), Ralph Ostermann (Danogips GmbH), Heinrich Rohlf's (Fermacell GmbH), Dr. Winfried Spickermann (Saint-Gobain Rigips GmbH)

Part 1 Technical parameters

Parameters (unit)	Determination method(s)	Sample pre-treatment	Remark / References
Particle size (mm)	Granulometry		Eurogypsum-QA FGD gypsum
Humidity (% w/w)	VGB serial number 1 or VGB serial number 3	None	Gravimetry quick dryer, drying cabinet 40°C or TGA
CaSO ₄ x 2 H ₂ O (% w/w)	VGB serial number 2.3 or VGB serial number 3	Pre-dried sample at 40-50°C to constant weight (odour determination at the same time)	Gravimetry (Sulfate determination) In sufficient quantity at 360°C in muffle kiln or quick test ultra-x at 360°C Methods to determine the calcium content are not recommended.
Organic carbon (% w/w TOC)	Digit 3.1.3.2 DepV DIN EN 13137		DIN EN 13137 DepV
Elimination of visible impurities	DIN EN 933-11 (10 kg sample, indication optic, visible components in % w/w)	Material > 1mm: Observe representative sample-taking	Method to classify components in coarsely recycled aggregates
Odour	VGB serial number 6	No odour at 40 - 50 °C (drying to constant weight)	Perception
Magnesium salts water soluble (% w/w MgO)	VGB serial number 8.1.2 or 8.7	VGB serial number 0.3 Stock solution A3	AAS or ICP OES
Sodium salts water soluble (% w/w Na ₂ O)	VGB serial number 8.2.2 or 8.7	VGB serial number 0.3 Stock solution A3	AAS or ICP OES
Potassium salts water soluble (% w/w K ₂ O)	VGB serial number 8.3.2 or 8.7	VGB serial number 0.3 Stock solution A3	AAS or ICP OES
Chloride (% w/w Cl)	VGB serial number 8.8 (VGB serial number 8.8.1, 8.8.2 or 8.8.3 or A 8.8)	VGB serial number 0.3 Stock solution A3	Potentiometry, ion chromatography or titration or photometric determination

Parameters (unit)	Determination method(s)	Sample pre-treatment	Remark / Reference
pH value	VGB serial number 4	if need be, pre-dry sample at 40-45° C in drying cabinet (VGB serial number 1.1)	Following DIN EN ISO 787-9: 1995-04

Part 2 Health Parameters (solid and fluoride)

Parameters (unit)	Determination method(s)	Sample pre-treatment	Remark / Reference
		DIN EN 13657	Aqua regia dissolution (HNO ₃ + HCl 1:3) as DepV with fermentation tube or in microwave (compulsory for Hh)
As (mg/kg)	VGB serial number 9 ICP OES (DIN EN ISO 11885)		DIN EN ISO 11885 Determination of selected elements ICP-OES (acc. to DepV)
Be (mg/kg)			
Pb (mg/kg)			
Cd (mg/kg)			
Cr (mg/kg)			
Co (mg/kg)			
Cu (mg/kg)			
Mn (mg/kg)			
Ni (mg/kg)			
Se (mg/kg)			
Te (mg/kg)			
Tl (mg/kg)			
V (mg/kg)			
Zn (mg/kg)			
Hg (mg/kg)	Digit 3.1.11 DepV		DIN EN 1483 AAS DIN EN 12338 Mercury - Process after enrichment by amalgamation DIN EN ISO 17852, atomic fluorescence spectrometry (acc. to MantelVO)
S elementary	Declaration „no odour of sulphur“.		Recorded with odour test
PAK (mg/kg)	DIN ISO 18287		Gas-phase chromatograph process with confirmation by mass spectrometry (GC-MS) (acc. to DepV)

Parameters (unit)	Determination method(s)	Sample pre-treatment	Remark / Reference
Fluoride (% w/w F)	Digit 3.2.16 DepV	VGB SERIAL NUMBER 0.3 Stock solution A3	DIN EN ISO 10304-1 (liquids ion chromatography) DIN 38405-4 anions (group D); determination g of fluoride (D 4)

Abbreviations:

VGB: Information Sheet Analysis FGD gypsum M701 <http://www.vgb.org> (2. issue 2008)

DepV: Landfill Ordinance Annex 4 Guidelines on sampling (taking of samples, preparation of samples and analysis of wastes and landfill replacement construction materials)

AAS Atomic absorption spectrometry
 ICP-MS Inductively coupled plasma mass spectrometry
 ICP-OES Inductively coupled plasma – optical emission spectrometry