



Association of German Agricultural Analytic and Research Institutes

EU FERTILISER RING TEST Q2/2010: DOLOMITE (Magnesium Calcium Carbonate)

In 2010, the Association of German Analytical and Research Institutes (VDLUFA e. V.) carried out an international fertiliser ring test to determine major and minor components of dolomite (magnesium calcium carbonate). Purpose of this fertiliser ring test was to offer a platform for testing and documenting the analytical quality of laboratories in all EU countries. 48 laboratories from 13 European countries took part in the ring test with the designation EU Q2/2010.

The analytes to be reported by the participating laboratories had to be determined by various official or standardized methods (see Table 1). Laboratories were asked to prepare the samples for analysis according EN 1482-2 (2007): "Fertiliser and liming materials - Sampling and sample preparation - Part 2: Sample preparation". Four aliquots of the sample material of the ring test EU Q2/2010 were prepared in order to be analysed. The results obtained for each individual determination (n=4) were reported in the units (mass % resp. mg/kg) given in Table 1, based on fresh mass (fm) respectively, with respect to the elements Cd, Pb, Cr, Ni, Hg and As, and on dry mass (dm).

Table 1: Analytes to be determined and methods used

No	Analyte	Method Digestion / Extraction	Method Final determination	Unit	Comments
1	calcium and magnesium	EN*) 12946:2000	EN 12946:2000	mass % fm	reported as Ca and Mg
2	calcium	EN 12947	VDLUFA*) Methods Book II.1, No 6.2.4 or 4.2.4	mass % fm	reported as Ca
3	magnesium	EN 12947	EN 12947, VDLUFA Methods Book II.1, 4.2.4	mass % fm	reported as Mg
4	size distribution	EN 12948	EN 12948	mass % fm	reported as 3,15 mm and 1,00 mm screened fraction after dry sieving
5	moisture content	EN 12048	EN 12048	mass % fm	reported as moisture content
6	reactivity	EN 13971 : 2008	EN 13971 : 2008	mass % fm	reported as reactivity
7	neutralisation value	EN 12945 :2008	EN 12945 :2008	mass % fm	reported as CaO
8	Cd-aqua regia	ISO 11466:1995	ICP-OES	mg/kg dm	reported as Cd
9	Pb-aqua regia	ISO 11466:1995	ICP-OES	mg/kg dm	reported as Pb
10	Cr-aqua regia	ISO 11466:1995	ICP-OES	mg/kg dm	reported as Cr
11	Ni-aqua regia	ISO 11466:1995	ICP-OES	mg/kg dm	reported as Ni
12	Hg-aqua regia	ISO 11466:1995	CV-AAS or AFS	mg/kg dm	reported as Hg
13	As-aqua regia	ISO 11466:1995	Hydride-AAS	mg/kg dm	reported as As

*) EN 1482-2 (2007): Fertiliser and liming materials - Sampling and sample preparation, Part 2: Sample preparation

**) VDLUFA (Ed., 1995-2012): VDLUFA-METHODS BOOK, Volume II.1, Fertiliser Analysis

Sample extraction according to ISO 11466 (aqua regia extraction in an open system) was strongly recommended for the analysis of Cd, Pb, Ni, Cr, Hg, and As. For the final determination of Cd, Pb, Ni and Cr, ICP-OES (inductive coupled plasma optical emission spectrometry) was recommended. Any other instrumental method (e. g. AAS, ICP-MS) was accepted. Concerning the final determination of Hg, cold-vapour AAS or AFS and for As hydride AAS was recommended. The statistical evaluation was done by robust methods (DIN 38402 A45, Q-method, HAMPEL estimate). Z_u -scores (tolerance limit $|Z_u| \leq 2,0$) were calculated as a bias estimate using IUPAC guidelines, so that laboratories can evaluate their performance in comparison to other laboratories. HorRat values were calculated for the methods in case a sufficient number of results had been reported. For all statistical calculations, the validated software package ProLab was used.

Table 2 shows all mean values, comparative standard deviations (absolute + relative), repeated standard deviation, tolerance limits and HorRat values.

Interested laboratories can be supplied with material from the tested fertiliser in order to use it as internal reference material (see order form).

Mean, Standard Deviation, HorRat and Tolerance Limits

Sample	Measurand	Unit	Mean Value	Reprod.S.D.	Rel.Reprod.S.D.	Repeat.S.D.	Rel.Repeat.S.D.	HorRat	Lower Tol.	Upper Tol.	Laboratories	Values	Stat.Meth.
DOLOMIT	CAEN946	mass%	20,622	0,683	3,310	0,090	0,44	1,305	19,280	22,010	20	78	DIN38402
DOLOMIT	MGEN946	mass%	11,647	0,365	3,136	0,083	0,71	1,134	10,930	12,390	21	82	DIN38402
DOLOMIT	CAVDLUF	mass%	20,729	1,180	5,691	0,168	0,81	2,245	18,430	23,160	36	132	DIN38402
DOLOMIT	MGEN947	mass%	11,742	0,523	4,452	0,101	0,86	1,613	10,720	12,810	36	132	DIN38402
DOLOMIT	SIZE315C	mass%	0,362	0,233	64,231	0,091	25,18		0,060	0,990	27	108	DIN38402
DOLOMIT	SIZE100C	mass%	20,117	1,584	7,876	0,494	2,46		17,060	23,420	30	120	DIN38402
DOLOMIT	MOISTURE	mass%	1,603	0,216	13,489	0,045	2,83		1,200	2,070	37	140	DIN38402
DOLOMIT	REACT	mass%	15,069	1,779	11,803	0,370	2,45		11,700	18,860	18	72	DIN38402
DOLOMIT	NEUTRAL	mass%	54,857	0,921	1,678	0,168	0,31	0,767	53,030	56,710	24	96	DIN38402
DOLOMIT	CD	mg/kg	0,649	0,156	23,954	0,018	2,77	1,403	0,370	1,010	41	150	DIN38402
DOLOMIT	PB	mg/kg	19,888	5,795	29,136	0,415	2,09	2,856	9,560	33,720	42	154	DIN38402
DOLOMIT	CR	mg/kg	6,208	2,012	32,418	0,153	2,47	2,667	2,660	11,120	40	146	DIN38402
DOLOMIT	NI	mg/kg	3,498	1,259	35,992	0,101	2,90	2,717	1,320	6,640	40+1	146	DIN38402
DOLOMIT	HG	mg/kg	0,011	0,009	78,758	0,001	6,40	2,508	0,002	0,035	24+10	88	DIN38402
DOLOMIT	AS	mg/kg	12,202	2,485	20,361	0,312	2,56	1,855	7,640	17,790	36	130	DIN38402