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Report on

Reference materials in adequate quantity (Part of D 4.1)

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Workpackage:	WP4, Preparation and improvement of MEAs and components
Workpackage leader (partner):	Tomas Klicpera (Fuma-Tech)
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PU	Public	
PP	Restricted to other programme participants (including the Commission Services)	
RE	Restricted to a group specified by the consortium (including the Commission Services)	
CO	Confidential, only for members of the consortium (including the Commission Services)	X

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MEA PRECURSORS

BENCHMARK MEAS

This document summarises the precursor materials used in the preparation of the first 104 5-layer benchmark MEA¹s (Table 1) prepared and tested within the KeePEMAlive project. The electrodes of these MEAs are prepared by IRD using an ultrasonic spray-coating technique either directly onto the membrane² as in the MEA batch from Jun/Jul-10 (Table 1) or onto the GDL³ as for the provided MEAs in Nov-10. The MEA precursors used for the benchmark MEAs are listed in Table 2.

The first batch of benchmark MEAs show an initial acceptable performance, but an un-acceptable durability as membrane failure was developed rather quickly. The reason was partly due to the applied lamination procedure, which was optimised for Nafion and not FumaPEM membranes. Much better durability was obtained by all Partners in the second MEA batch provided.

Table 1 Summary of manufactured benchmark MEAs 2010.

Partner	Active Electrode dimension [cm x cm]	Membrane Dimension [cm x cm]	Qty of MEAs Jun/Jul-10	Qty of MEAs Nov-10	Total Qty of MEAs 2010
IRD	1.7 x 1.7	2.5 x 2.5	0	0	0
	2.55 x 2.55	3.5 x 3.5	8	8	16
ECN	∅ = 3.1	7.0 x 7.0	3	8	11
	7.1 x 7.1	12.7 x 12.7	2	6	8
Fumatech	5.0 x 5.0	7.0 x 7.0	2	0	2
	7.1 x 7.1	10.0 x 10.0	0	0	0
SINTEF	5.0 x 5.0	8.0 x 8.0	2	3	5
	2.3 x 2.3	4.3 x 4.3		5	5
Graz	12.0 x 2.2	13.5 x 3.5	3	8	11
Eifer	5.0 x 5.0	7.0 x 7.0	2	10	12
JRC	5.0 x 5.0	7.5 x 7.5	0	3	3
	2.3 x 2.3	5.0 x 5.0	0	3	3
CNRS	1.7 x 1.7	3.7 x 3.7		10	10
	2.55 x 2.55	4.6 x 4.6	8	10	18
Total number of MEAs			30	74	104
Total electrode area (cm ²)			456	1,618	2,074
Total membrane area (cm ²)			1,203	4,428	5,631

¹ MEA: Membrane Electrode Assembly

² CCM: Catalyst Coated Membrane

³ GDL: Gas Diffusion Layer

CCB: Catalyst Coated Backing equivalent to GDE: Gas Diffusion Electrodes

Table 2 The MEA precursors and catalyst loadings for the 5-layer benchmark MEAs.

Description	Product ID	Catalyst loading	Data sheet	Comments
Anode GDL	Sigracet 35DC		Annex 4	The DC grade refers to a PTFE ⁴ content of 20%
Anode Catalyst	62RKR4	0.3 mg PtRu/cm ²	Annex 2 & 3	
Membrane	Fumapem F-940		Annex 5	
Cathode Catalyst	65KR2	0.5 mg Pt/cm ²	Annex 1 & 3	
Cathode GDL	Sigracet 35DC		Annex 4	Do Anode GDL

MEAS BASED ON IMPROVED PRECURSORS

Experimental MEAs based on a reinforced FumaPEM membrane (Annex 6) has been tested with promising results for almost 400 hours by IRD (Fig. 1). The results are so encouraging that the following MEA batches will be based on this membrane.

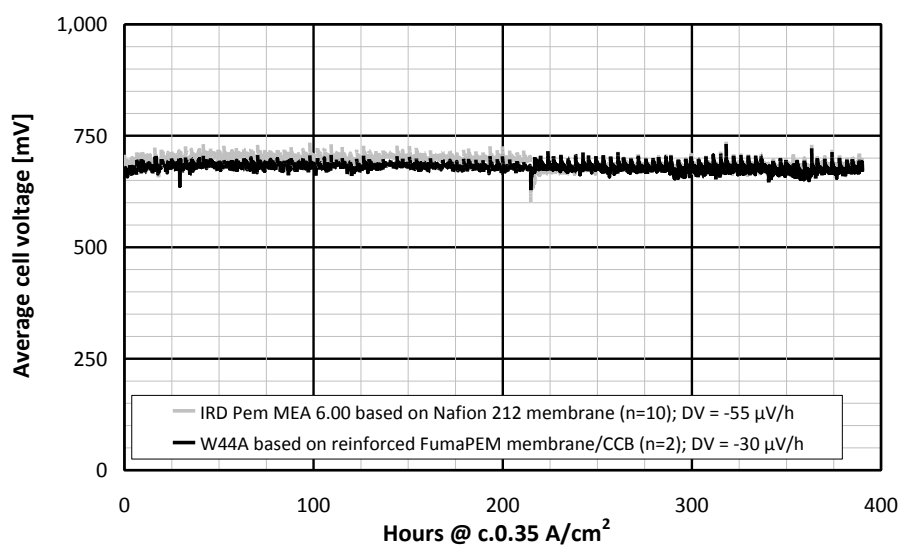


Fig. 1

Longer-time performance of the new KeepPEMAlive MEA equipped with reinforced FumaPEM membrane compared to the IRD *state-of-the-art* MEA based on Nafion membrane.

⁴ PTFE: PolyTetraFluoroEthylen also known as Teflon

ANNEX 1: BENCHMARK CATHODE CATALYST SPECIFICATION



Superior
MicroPowders

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From: Cabot Superior Micro Powders
5401 Venice Ave NE
Albuquerque, NM 87113
USA

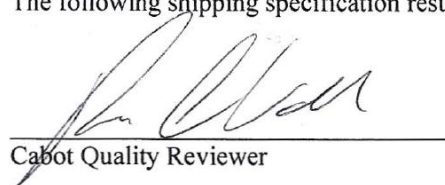
To: IRD Fuel Cells LLC
8500 Washington, Building B
Albuquerque, NM 87113
USA

Customers Order:
Product: Pt on Carbon Cathode 65KR2
Batch/Lot Number: AQ706086

Delivery Number: 007
Date of Print: Jan. 15, 2010

Certificate of Quality and Analysis

The catalyst powder shipped in this order has been manufactured in accordance with our manufacturing operating procedures and meets our manufacturing operating standards.
The following shipping specification results on the above product are provided for your knowledge and use.



Cabot Quality Reviewer

Product Data:

Catalyst Description: 65 wt% Pt/Ketjen Black EC600

Grams of Catalyst: 60

Characteristic	Value	Unit	Specification	
			Min	Max
BET	308	m ² /g	300	N/A
XRD (RIQAS) % Amorph	69	%	65	80
XRD (RIQAS) Amorph Size	2.9	nm	2.8	4.0
PSD d50	7.1	microns	6.3	8.6
PSD d90	16.6	microns	14.4	19.0
Pt Assay	65	%	61	70
Powder Metal Ion Impurity (Al,Ca,Cu,Fe,Co,Cr,Mg,Ni,Pb,Pd,Ag)	176	ppm		400
Carbon Support (Al,Ca,Cu,Fe,Co,Cr,Mg,Ni,Pb,Pd,Ag)	110	ppm		150

ANNEX 2: BENCHMARK ANODE CATALYST SPECIFICATION



Superior
MicroPowders

Page 1 of 1

From: Cabot Superior Micro Powders
5401 Venice Ave NE
Albuquerque, NM 87113
USA

To: IRD Fuel Cells LLC
8500 Washington, Building B
Albuquerque, NM 87113
USA

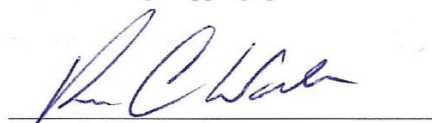
Customers Order:
Product: PtRu on Carbon Anode 62RKR4
Batch/Lot Number: AQ10063EX

Delivery Number : 009
Date of Print: March 31, 2010

Certificate of Quality and Analysis

The catalyst powder shipped in this order has been manufactured in accordance with our manufacturing operating procedures and meets our manufacturing operating standards.

The following shipping specification results on the above product are provided for your knowledge and use.



Cabot Quality Reviewer

Product Data:

Catalyst Description: 62 wt% PtRu/Ketjen Black EC600

Grams of Catalyst: 80

Characteristic	Value	Unit	Specification	
			Min	Max
BET	346	m ² /g	306	391
PSD d50	5.6	microns	5.4	6.2
PSD d90	15.0	microns	14.2	17.6
XRD 2 theta angle	40.4	Degree	40.3	40.5
XRD FWHM	2.4	Degree	2.3	2.6
PtRu Assay	62.4	%	59	65
Pt Assay	41.4	%	40	43
Ru Assay	20.9	%	19	22
Pt/Ru Atomic Ratio	1.02		0.97	1.10
Powder Metal Ion Impurity (Al,Ca,Cu,Fe,Co,Cr,Mg,Ni,Pb,Pd,Ag)	234	ppm		600
Carbon Support (Al,Ca,Cu,Fe,Co,Cr,Mg,Ni,Pb,Pd,Ag)	110	ppm		150

ANNEX 3: BENCHMARK CATALYST SUPPORT SPECIFICATION

Product Data Sheet



AkzoNobel
Tomorrow's Answers Today

Ketjenblack® EC-600JD

Chemical description	Electroconductive carbon black, pellets
	CAS No. : 1333-86-4 EINECS/ELINCS No. : 215-609-9 TSCA status : listed on inventory
Specifications	Appearance : Black free-flowing pellets Pore volume (DBP) : 480-510 ml/100 g Fines smaller than 125 micron : 7% max. Grit content : 30 mg/kg max. Moisture : 0.5% max. Volatiles : 1.0% max. Iodine adsorption : 1000-1100 mg/g Ash : 0.1% max. pH : 8-10 Apparent bulk density : 100-120 kg/m ³
Storage	AkzoNobel recommends to store <i>Ketjenblack</i> EC-600JD in a dry place away from direct sunlight. The packaging can be damaged if exposed to direct sunlight for more than 30 minutes. When stored under these recommended storage conditions, <i>Ketjenblack</i> EC-600JD will remain within the AkzoNobel specifications for a period of at least six months after delivery.
Packaging and transport	The standard packaging is 8 kg net in a vented PE bag. The vent consists of a small plastic valve which contains HDPE, NBR and cellulosic parts, which are not readily dispersed during compounding. Due to this we strongly advice not to put the <i>Ketjenblack</i> EC-600JD including the bag into the mixing system. A full pallet carries 320 kg net. Both packaging and transport meet the international regulations. For the availability of other packed quantities contact your AkzoNobel representative. <i>Ketjenblack</i> EC-600JD is classified as a non-hazardous good according to national and international transport regulations.
Safety and handling	Please refer to the Material Safety Data Sheet (MSDS) for further information on the safe storage, use and handling of <i>Ketjenblack</i> EC-600JD. This information should be thoroughly reviewed prior to acceptance of this product. The MSDS is available at www.akzonobel.com/polymer .

PA 10383.07/August 2008
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Applications

Ketjenblack EC-600JD is a very pure carbon black extremely suitable for antistatic and electroconductive applications. Due to its unique morphology and the extremely high surface area of approx. 1400 m²/g (BET), only one sixth the amount of *Ketjenblack* EC600-JD is needed compared to conventional electroconductive blacks in order to achieve the same conductivity. The extreme low loading of *Ketjenblack* EC600-JD allows easier processing for those compounds sensitive to filler addition, thus minimizing loss in mechanical and rheological properties.

Ketjenblack EC-600JD when thoroughly dispersed with the polymer significantly increases the conductivity of the resulting compound.

Ketjenblack EC-600JD can be used in all types of polymers, thermoset, thermoplastic as well as elastomers. Due to its unique morphology and structure with very low amounts of *Ketjenblack* EC-600JD excellent conductive material can be made. The loading needed to obtain a certain conductivity can vary significantly per type of polymer. More detailed information is available in the special *Ketjenblack* EC Technical Bulletin.

Ketjenblack EC-600JD has a low ash content, which in addition with the extreme low loading level, makes it an excellent product for semicon applications in cable shielding. The low grit content provides very smooth surface during extrusion.

Ketjenblack EC-600JD can also be used to produce conductive coatings and primers. Also here already at very low loading levels optimal electroconductive performance is obtained, which even more minimizes loss in mechanical and rheological properties compared to *Ketjenblack* EC-300J. With *Ketjenblack* EC-300J about double the amount is needed to obtain the same conductivity

Some other applications are batteries, packaging for IC parts, tubing, flooring, carpet backing, automotive parts, cell phones and many more.

Ketjenblack is a registered trademark of Akzo Nobel Chemicals B.V. or affiliates in one or more territories.

All information concerning this product and/or suggestions for handling and use contained herein are offered in good faith and are believed to be reliable. AkzoNobel Polymer Chemicals, however, makes no warranty as to accuracy and/or sufficiency of such information and/or suggestions, as to the product's merchantability or fitness for any particular purpose, or that any suggested use will not infringe any patent. Nothing contained herein shall be construed as granting or extending any license under any patent. Buyer must determine for himself, by preliminary tests or otherwise, the suitability of this product for his purposes. The information contained herein supersedes all previously issued bulletins on the subject matter covered. The user may forward, distribute, and/or photocopy this document only if unaltered and complete, including all of its headers and footers, and should refrain from any unauthorized use. You may not copy this document to a website.

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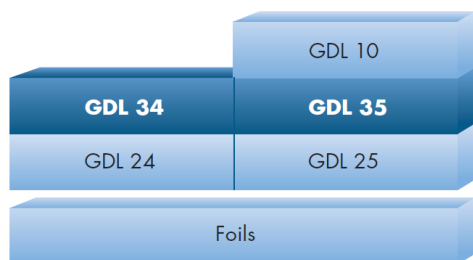
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ANNEX 4: BENCHMARK GDL SPECIFICATION

SIGRACET® GDL 34 & 35 Series Gas Diffusion Layer



After the introduction of our 300-micron thick GDL series GDL 30/31, SGL Group has been further improving the series with respect to production yield and product variability. We have introduced our newest grades, GDL 34 and GDL 35, to the commercial market. SGL Group has invested heavily in this product development and has been contributing resources from a number of different and cross-functional parts of the organization. The result has been the launch of our new grades GDL 34/35.

Users have confirmed the excellent match of the properties:

- Greatly improved homogeneity inter-lot and intra-lot. The overall variability has been shown to be better than +/- 10% (2 sigma)

- Greatly improved surface flatness
- Greatly reduced occurrence of faulty spots on a roll
- Greater roll length
- Best performance under various operating conditions

The above list is not sacrificing any of the list of properties which makes SGL Group's GDL unique:

- Produced and shipped as a continuous roll good
- Utilizing low-cost nonwoven processes
- Hydrophobic treatment of the substrate to the desired level by coating the bulk surfaces with PTFE
- Coating of material grades with SGL Group's unique microporous layer for better membrane contact, water management and mechanical protection
- Concept of lower and higher porosity (GDL 34: lower, GDL 35: higher porosity) to react with cell humidity level.

The introduced product series GDL 34/35 is intended to combine required production robustness and excellent performance properties. Stack developers and assemblers are benefiting from the greatly improved characteristics.

Properties of SIGRACET® GDL 34/35

Property	Unit	GDL 34 BA	GDL 34 BC	GDL 35 BA	GDL 35 BC
Thickness ▲	mil	11.0	12.4	11.8	12.7
	µm	280	315	300	325
Areal Weight	oz/ft ²	0.28	0.46	0.18	0.36
	g/m ²	86	140	54	110
Air Permeability ■	cm ³ /(cm ² · s)	45	0.35	170	1.5
Electrical Resistance (through plane) ●	mΩ cm ²	< 11	< 14	< 12	< 15




- ▲ Under 3,45 N/cm² (5psi), sample diameter 30 mm
- Gurley model 4118, 300 cc, 0.1 sq.in orifice, 304 Pa
- 2-point measurement, circular (25 mm diameter), gold plated contacts, 100 N/cm²

Broad Base. Best Solutions.



Grades

We supply SIGRACET Gas Diffusion Layers in roll form in lengths of 75 +/- 25 m. Our standard roll width is 45 cm, but we can split parent rolls into multiples thereof, i. e. 2 x 22.5 cm wide rolls, 3 x 15 cm wide rolls, etc. Sheets stamped to a specified geometry are also available if so desired. Substrate PTFE loadings are available from 0 to 30 wt% whereas 5 wt% is standard.

	AA	YA	YC			
						
	Y	A	B	C	D	E
% PTFE		0	5	10	20	30

GDL "AA" is our plain substrate with no value-added post-processing.
GDL "BA" is our hydrophobized substrate with a 5 wt% PTFE loading.
GDL "BC" is our hydrophobized substrate (5 wt% PTFE) with our standard Microporous Layer (MPL) on one side.



By courtesy of Viessmann



By courtesy of P21

Unique Characteristics

Our GDL 34/35 are of a 2-dimensional structure designed to have lower thickness, higher bending stiffness, lower compression set, and lower compressibility than GDL 10.

GDL 34/35 has also greatly improved tolerances (thickness and areal weight) and a lower anisotropy factor between the machine and transverse directions.

On request, we will advise you on maximum compression loads for use in bonding to Catalyst-Coated Membranes (CCMs) or assembling stacks. Gasket design also needs to take the compression behavior into account.

® registered trademark of SGL Group companies

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This information is based on our present state of knowledge and is intended to provide general notes on our products and their uses. It should therefore not be construed as guaranteeing specific properties of the products described or their suitability for a particular application. Any existing industrial property rights must be observed. The quality of our products is guaranteed under our "General Conditions of Sale".

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SGL GROUP
THE CARBON COMPANY

ANNEX 5: BENCHMARK MEMBRANE SPECIFICATION

Technical Datasheet - fumapem[®] F



fumapem[®] F

General

fumapem[®] F membranes are based on perfluorinated sulfonic acid / PTFE copolymers with excellent chemical stability and superior ionic conductance intended for use in fuel cell applications or electrolysis.

Membranes are identified by membrane type and identification number (Lot-No.). Please refer to this type and identification number in case of queries.

Delivery

Membranes are delivered on / between backing film(s) (PET) and either ready to use (pre-treated, activated) or non-activated. In non-activated case the membrane must be activated before use. Depending on the MEA preparation technique, activation can be applied before or after MEA preparation (see below).

Handling

Keep membrane package closed / sealed when unused. Unpack membrane only for direct use and process immediately after opening. Store, handle and process the membrane in a clean and dust-free area. Use only new and sharp knives or blades, when cutting the membrane.

Always wear protective gloves when handling the membrane.

Handle with care, be sure not to bend, kink, fold, puncture, crease or scratch the membrane, otherwise cracks or leaks will occur. All surfaces which may get into contact with the membrane during inspection, storage, processing, pretreatment and mounting must be free of sharp edges or angles.

Pretreatment / Activation (for non-activated membranes only)

For optimum performance and lowest resistance it is necessary to pretreat membranes according to the following prescription: Put the membrane / MEA sample in an aqueous 10 wt% HNO₃ solution (or in 5 vol % H₂SO₄ solution) for at least 12 h at T = 80 °C. After treatment in demineralised water at T = 80 °C for 1 h, and after rinsing with demineralised water (pH ~ 7) the membrane / MEA is ready for use.

If you have any concerns about storage, chemical stability and pretreatment before proceeding, please feel free to contact us for further information.



FuMA-Tech GmbH

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Technical Datasheet - fumapem[®] F

Physical and chemical data of fumapem[®] F membranes

fumapem [®]		F-950	F-940	F-930
IEC	meq g ⁻¹	1.05 – 1.11	1.05 – 1.11	1.05 – 1.11
EW	g eq ⁻¹	900 – 950	900 – 950	900 – 950
thickness (dry)	µm	50	40	30
water uptake in H ₂ O at 25 °C ^{a)}	wt %	32 – 35	30 – 35	30 – 32
dimensional swelling in H ₂ O at 25 °C ^{b)}	%	17 – 19	15 – 20	15 – 17
conductivity ^{c)}	mS cm ⁻¹	90 – 100	90 – 100	90 – 100
area resistance ^{c)}	Ω cm ²	0.05	0.04	0.03
methanol permeation ^{d)}	Mol h ⁻¹ m ⁻²	120	-	200
Young's modulus at 23 °C / 50 % r.h. ^{e)}	MPa	200 – 250	200 – 250	200 – 250
yield strength at 23 °C / 50 % r.h. ^{e)}	MPa	9 – 11	9 – 11	9 – 11
tensile strength at 23 °C / 50 % r.h. ^{e)}	MPa	> 20	> 20	> 20
elongation at break at 23 °C / 50 % r.h. ^{e)}	%	> 220	> 220	> 200
density (dry)	g cm ⁻³	1.98 – 2.01	1.98 – 2.01	1.98 – 2.01
start of thermal decomposition ^{f)}	°C	> 300	> 300	> 300
glass transition temperature T _g ^{g)}	°C	110	110	110

a) reference membrane dried over P₂O₅ in vacuo.

b) reference membrane dried at room temperature and 50 % r.h.

c) in H⁺-form at T = 25 °C in H₂O

d) determined in a concentration cell (1.0 M methanol versus deionized water at T = 50 °C).

e) according to DIN EN 527-1.

f) in H⁺-form and dry state using TGA (heating rate 2 K min⁻¹).

g) in H⁺-form and dry state using DMA (heating rate 2 K min⁻¹, frequency 1 Hz), maximum of tanδ.

Please note: The data are not measured directly on the item supplied. The table contains membrane types with standardized equivalent weights and thicknesses. Supplied samples may differ in thickness and equivalent weight.

FuMA-Tech GmbH

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