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ZLG-P-429.08.10

TEST REPORT

22.01.2010

Test report no. Z09ML937SI

Evaluation of the effectiveness of

Germ Free 24

Test virus: Influenza A virus H1N1 (swine)

Method: following EN 14476:2007-02

Sponsor:

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1. Introduction

The objective of this study was to evaluate the virus-inactivating properties of the hand disinfectant Germ Free 24 against influenza A virus H1N1 (swine) using a quantitative suspension assay following EN 14476:2007-02 (1).

2. Test laboratory

MikroLab GmbH, Norderoog 2, D-28259 Bremen

3. Identification of the sample

Manufacturer	Zoono Ltd.
Name of product	Germ Free 24
Application	hand hygiene
Batch number	-
Expiry date	-
Active compound (s)	-
Appearance and odour	clear, colourless liquid; product specific
pH-value (s)	undiluted: 4.86 (20°C)
Storage conditions	room temperature in the dark (area with restricted access)
Date of arrival in the laboratory	19.10.2009

4. Materials

4.1 Culture medium and reagents

- Eagle`s Minimum Essential Medium with Earle`s BSS (EMEM, Lonza Group Ltd., catalogue no. BE12-125F)
- Fetal calf serum (Biochrom AG, article no. S 0115)
- 1.4 % Formaldehyde solution (Chemisch-technologisches Laboratorium Dr. Melzer, D-28199 Bremen)
- Aqua bidest. (Fresenius Kabi Deutschland, article no. P2N 1636071)
- PBS (Invitrogen, article no. 18912-014)

4.2 Virus and cells

The influenza A virus sw/Greven/IDT2889/2004 H1N1 virus was obtained from Prof. Dr. Georg Herrler, Institute of Virology at the School of Veterinary Medicine Hannover (Tierärztliche Hochschule, D-30559 Hannover). This virus strain was introduced in this study as surrogate of the pandemic strain influenza A virus /California/04/2009 H1N1 due to bio safety reasons.

The *MDCK cells* were obtained from the Friedrich-Löffler-Institut, Bundesforschungsinstitut für Tiergesundheit (formerly Bundesforschungsanstalt für Viruskrankheiten der Tiere, isle of Riems) (Dr. R. Riebe, catalogue no. RIE 244).

The cells were inspected regularly for morphological alterations and for contamination by mycoplasmas. No morphological alterations of cells and no contamination by mycoplasmas could be detected.

4.3 Apparatus, glassware and small items of equipment

- CO_2 incubator (Nunc GmbH & Co. KG, model QWJ 350)
- Agitator (Vortex Genie Mixer, type G 560E)
- pH measurement 315i (WTW, article no. 2A10-100)
- Centrifuge (Sigma-Aldrich-Chemie GmbH, type 113)
- Microscope (Olympus, type CK 30)
- Water bath (JULABO, Julabo U 3)
- Adjustable volume automatic pipettes (Eppendorf AG)
- Polysterol 96-well microtiter plate (Nunc GmbH & Co. KG, Wiesbaden, Germany)
- Cell culture flask (Nunc GmbH & Co. KG, Wiesbaden, Germany)
- Sealed test tubes (Sarstedt AG & Co., Nümbrecht, Germany)
- MicroSpin™ S-400 HR columns (GE Healthcare, Freiburg, Germany)

5. Experimental conditions

Test temperature	20°C ± 1.0°C
Concentration of test product	undiluted (80.0 %) and 10.0 % solutions (non-active range)
Contact times	30 and 60 seconds
Interfering substance	PBS
Diluent	water of standardised hardness (10.0 % solution)
Procedure to stop action of product	immediate dilution and gel filtration
Test virus strain	influenza A virus sw/Greven/IDT2889/2004 H1N1
Test period	19.10.2009 – 22.01.2010
End of testing	22.01.2010

6. Methods

6.1 Preparation of test virus suspension

To prepare the test virus suspension, *MDCK cells* that had been cultured with Eagle´s minimum essential medium (EMEM) and 10 % or 2 % fetal calf serum (FCS) were inoculated with swine influenza A virus in 175 cm^2 cell culture flasks. Once a cytopathic effect had been induced (approx. 24 hours), freezing and thawing was carried out once. The cell debris was removed by centrifugation at 3.000 rpm for ten minutes (4°C) and the supernatant was recovered as test virus suspension and stored in aliquots at -80°C.

6.2 Disinfectant

The test product was evaluated undiluted. Due to the addition of test virus suspension and PBS an 80.0 % solution resulted. The product was also tested as 10.0 % solution (demonstration of non-active range).

The 10.0 % solution was prepared with water of standardised hardness immediately before the inactivation tests.

6.3 Infectivity assay

Infectivity was determined by means of end point dilution titration using the microtitre process. For this, 100 µl aliquots of the samples, which had been serially diluted with ice-cold EMEM, were transferred to eight wells of a sterile polystyrol 96-well microtitre plate with a

preformed monolayer of *MDCK cells* (placed in each well on the previous day; 100 µl aliquots with approx. 1.5 x 10^4 cells). Incubation took place at 37°C in a CO_2 incubator (5 % CO_2 content) for five days. Finally, cultures were observed for cytopathic effects with a reversed microscope and the infective dose $TCID_{50}$/ml was calculated with the method of Kärber (2) and Spearman (3) with the following formula:

$$- \log_{10}TCID_{50} = X_0 - 0.5 + \sum r/n$$

meaning

X_0 = $\log_{10}$ of the lowest dilution with 100 % positive reaction

r = number of pos. determinations of lowest dilution step with 100 % positive and all higher positive dilution steps

n = number of determinations for each dilution step.

6.4 Calculation and verification of virucidal activity

The virucidal activity of the test disinfectant was evaluated by calculating the decrease in titre in comparison with the control titration without disinfectant. The difference is given as reduction factor (RF).

According to EN 14476:2007-02, a disinfectant or a disinfectant solution at a particular concentration is having virus-inactivating efficacy if the titre is reduced at least by four $\log_{10}$-steps within the recommended exposure period.

6.5 Inactivation assays

Investigations for determination of virucidal activity followed to EN 6.6. The test product was examined as undiluted (80.0 %) and 10.0 % solutions in hard water according to EN 5.2.2.2.
30 and 60 seconds were chosen as contact times.
Due to a more convenient handling, the volumes in this assay were 0.1 ml test virus suspension, 0.1 ml interfering substance and 0.8 ml test product. Immediately at the end of a chosen contact time, activity of the disinfectant was stopped by dilution to 10^{-8}.
A control of efficiency for suppression of test product activity was included (EN 6.6.6).
Titration of the virus control was performed at contact times 0 min and 60 min (EN 6.6.8).

Since the cytotoxicity did not allow following the reduction of residual infectivity titre over the range of four $\log_{10}$-steps, ready to use MicroSpin™ S-400 HR columns were used in order to remove the cytotoxic agents according to the instructions of the manufacturer. Virus controls with and without MicroSpin™ S-400 HR columns were included.

It must be pointed out that values with columns are only applicable when no residual virus can be found. This is related to the fact that in some cases virus may remain in the columns which would lead to a wrongful advantage for the disinfectant. Small amount of remaining virus can be a hint that values can be regarded as being borderline cases and thus not active.

Furthermore, a cell control (only addition of medium) was incorporated.

Inactivation tests were carried out in sealed test tubes in a water bath at 20°C ± 1.0°C. Aliquots were retained after appropriate exposure times, and residual infectivity was determined.

6.6 Determination of cytotoxicity

Determination of cytotoxicity was performed according to EN 6.6.4.1 with 200 µl hard water and 800 µl test product.
Values are given as $\log_{10}CD_{50}$/ml (in analogy to $\log_{10}TCID_{50}$/ml).

6.7 Cell sensitivity to virus

For the control of cell sensitivity to virus two parts by volume hard water were mixed with eight parts by volume of the product. This mixture was added to a MicroSpin™ S-400 HR column and diluted after centrifugation and transferred to the cells.
Finally, a comparative titration of the test virus suspension was performed on the treated and non-treated cells

6.8 Control of efficacy for suppression of test product activity

Furthermore, a control of efficiency for suppression of test product activity was included (EN 6.6.6).

6.9 Reference virus inactivation test

As reference for test validation a 0.7 (w/v) % formaldehyde solution according to EN 6.6.7.1 was included. Contact times were 5 and 15 minutes. In addition, cytotoxicity of formaldehyde test solution was determined following EN 6.6.7.2 with dilutions up to 10^{-5}.

7. Verification of the methodology

The following criteria as mentioned in EN 8.3 were fulfilled:

a) The titre of the test virus suspension allowed the determination of a 4 $\log_{10}$-reduction after gel filtration.

b) The cytotoxicity of the test product (80.0 % solution) was two $\log_{10}$ steps. After treatment with columns the cytotoxicity was reduced to ≤1.50.

c) The comparative titration on pretreated (test product) and non pretreated (PBS) *MDCK cells* showed an acceptable difference (<1 $\log_{10}$; EN 8.3) of virus titres: 6.38 (PBS) versus 5.75 (disinfectant) $\log_{10}$ $TCID_{50}$/ml.

d) The control of efficacy for suppression of disinfectant activity showed a decrease in virus titre due to the fact that even the 10.0 % solution was slightly active.

Since all criteria following EN 8.3 were fulfilled, examination with influenza A virus H1N1 (swine) following EN 14476:2007-02 is valid.

8. Results

Results of examination are shown in tables 1 to 7. Tables 1 to 6 demonstrate the raw data, whereas table 7 gives a summary of results.

The undiluted test product was able to inactivate influenza A virus H1N1 (swine) after 60 seconds in this quantitative suspension test. At that time point, no influenza A virus H1N1 (swine) was detectable. The reduction factor was ≥ 2.88.

Without columns it was not possible to demonstrate a reduction of four $\log_{10}$ steps. After introducing the columns, the undiluted solution was able to achieve a reduction factor of ≥ 4.63 after 60 seconds exposure time. This corresponded to an inactivation of ≥ 99.99 %.

The 10.0 % solution also showed a slight activity against the influenza A virus H1N1 (swine) after 60 seconds of exposure time.

9. Summary

In summary, a sufficient reduction of virus titre can be achieved by Germ Free 24 undiluted after an exposure time of 60 seconds. Due to the lack of virological guidelines simulating practical conditions in Europe (phase 2, step 2 tests) the data of this quantitative suspension test lead to the recommendation to use the hand disinfectant Germ Free 24 for inactivation of influenza A virus H1N1 (swine) as follows:

undiluted 60 seconds

Bremen, 22.01.2010

- Dr. J. Steinmann -

10. Quality control

The Quality Assurance of the results was maintained by performing the determination of the virus-inactivating properties of the disinfectant in accordance with Good Laboratory Practice regulations:

1) Chemicals Act of Germany, Appendix 1, dating of 01.08 1994 (BGBI. I, 1994, page 1703). Appendix revised at 14. 05. 1997 (BGBI. I, 1997, page 1060).
2) OECD Principles of Good Laboratory Practice (revised 1997); OECD Environmental Health and Safety Publications; Series on Principles of Good Laboratory Practice and Compliance Monitoring – Number 1. Environment Directorate, Organization for Economic Co-operation and Development, Paris 1998.

The plausibility of the results was additionally confirmed by controls incorporated in the inactivation assays.

11. Recorders to be maintained

All testing data, protocol, protocol modifications, the final report, and correspondence between MikroLab GmbH and the sponsor will be stored in the archives at MikroLab GmbH.

12. Literature

1. EN 14476:2007-02: Chemical disinfectants and antiseptics – virucidal quantitative suspension test - Test method and requirements (phase 2, step 1)

2. Kärber, G.: Beitrag zur kollektiven Behandlung pharmakologischer Reihenversuche. Arch Exp Path Pharmak; 162, 1931, 480-487

3. Spearman, C.: The method of `right or wrong cases` (constant stimuli) without Gauss´s formulae.
Brit J Psychol; 2 1908, 227-242

Appendix:

Table 1: Raw data of Germ Free 24 (80.0 %) tested against influenza A virus H1N1 (swine) without columns

Table 2: Raw data of Germ Free 24 (80.0 %) tested against influenza A virus H1N1 (swine) with columns (1st assay)

Table 3: Raw data of Germ Free 24 (80.0 %) tested against influenza A virus H1N1 (swine) with columns (2nd assay)

Table 4: Raw data of Germ Free 24 (10.0 %) tested against influenza A virus H1N1 (swine)

Table 5: Control of efficacy for suppression of test product activity

Table 6: Raw data (influenza A virus H1N1 swine) for cell sensitivity with columns

Table 7: Summary of results with Germ Free 24 and influenza A virus H1N1 (swine)

Table 1 : Raw data of Germ Free 24 (80.0 %) tested against influenza A virus H1N1 (swine) without columns (quantal test; 8 wells) (2142)

Product	Concentration	Interfering substance	Contact time (min)	Dilutions (log_{10})								
				1	2	3	4	5	6	7	8	9
Test product	80.0%	PBS	0.25	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			0.5	tttt tttt	tttt tttt	0003 0000	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			1.0	tttt tttt	tttt tttt	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			2.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Test product cytotoxicity	80.0%	PBS	n.a.	tttt tttt	tttt tttt	0000 0000	0000 0000	0000 0000	n.d.	n.d.	n.d.	n.d.
Formaldehyde	0.7% (m/V)	PBS	5	tttt tttt	tttt tttt	tttt tttt	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			15	tttt tttt	tttt tttt	tttt tttt	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			30	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			60	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Formaldehyde cytotoxicity	0.7% (m/V)	PBS	n.a.	tttt tttt	tttt tttt	tttt tttt	0000 0000	0000 0000	n.d.	n.d.	n.d.	n.d.
Virus control	n.a.	PBS	0	4444 4444	4444 4444	4444 4444	4444 4444	3430 1344	0020 0000	0000 0000	0000 0000	n.d.
			60	4444 4444	4444 4444	4444 4444	4444 4444	4444 3430	0000 0000	0000 0000	0000 0000	n.d.

n.a. = not applicable
n.d. = not done

0 = no virus present; t = cytotoxic
1 to 4 = virus present (degree of CPE in 8 cell culture units) (wells of microtitre plates)

Table 2 (1st assay): Raw data of Germ Free 24 (80.0 %) tested against influenza A virus H1N1 (swine) with columns (quantal test; 8 wells) (2091)

Product	Concentration	Interfering substance	Contact time (min)	Dilutions (log_{10})								
				1	2	3	4	5	6	7	8	9
Test product	80.0%	PBS	0.25	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			0.5	4034 0014	0200 4000	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			1.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			2.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Test product cytotoxicity	80.0%	PBS	n.a.	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.	n.d.	n.d.	n.d.
Formaldehyde	0.7% (m/V)	PBS	5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			30	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			60	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Formaldehyde cytotoxicity	0.7% (m/V)	PBS	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Virus control	n.a.	PBS	0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			60	4444 4444	4444 4444	4444 4444	4444 4444	4444 4444	0400 0440	0000 0000	0000 0000	n.d.

n.a. = not applicable
n.d. = not done

0 = no virus present; t = cytotoxic
1 to 4 = virus present (degree of CPE in 8 cell culture units) (wells of microtitre plates)

Table 3 (2nd assay): Raw data of Germ Free 24 (80.0 %) tested against influenza A virus H1N1 (swine) with columns (quantal test; 8 wells) (2142)

Product	Concentration	Interfering substance	Contact time (min)	Dilutions (log_{10})								
				1	2	3	4	5	6	7	8	9
Test product	80.0%	PBS	0.25	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			0.5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			1.0	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			2.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Test product cytotoxicity	80.0%	PBS	n.a.	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.	n.d.	n.d.	n.d.
Formaldehyde	0.7% (m/V)	PBS	5	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			15	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			30	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			60	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Formaldehyde cytotoxicity	0.7% (m/V)	PBS	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Virus control	n.a.	PBS	0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			60	4444 4444	4444 4444	4444 4444	4444 4444	0233 0203	0000 0000	0000 0000	0000 0000	n.d.

n.a. = not applicable
n.d. = not done

0 = no virus present; t = cytotoxic
1 to 4 = virus present (degree of CPE in 8 cell culture units) (wells of microtitre plates)

Table 4: Raw data of Germ Free 24 (10.0 %) tested against influenza A virus H1N1 (swine) (quantal test; 8 wells) (2142)

Product	Concentration	Interfering substance	Contact time (min)	Dilutions (log_{10})								
				1	2	3	4	5	6	7	8	9
Test product	10.0%	PBS	0.25	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			0.5	tttt tttt	4444 4444	3342 3344	4020 3140	0020 4000	0000 0000	0000 0000	0000 0000	n.d.
			1.0	tttt tttt	4433 3442	4133 3434	0040 0012	0000 0010	0000 0000	0000 0000	0000 0000	n.d.
			5.0	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Test product cytotoxicity	10.0%	PBS	n.a.	tttt tttt	0000 0000	0000 0000	0000 0000	0000 0000	n.d.	n.d.	n.d.	n.d.
Formaldehyde	0.7% (m/V)	PBS	5	tttt tttt	tttt tttt	tttt tttt	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			15	tttt tttt	tttt tttt	tttt tttt	0000 0000	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
			30	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
			60	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Formaldehyde cytotoxicity	0.7% (m/V)	PBS	n.a.	tttt tttt	tttt tttt	tttt tttt	0000 0000	0000 0000	n.d.	n.d.	n.d.	n.d.
Virus control	n.a.	PBS	0	4444 4444	4444 4444	4444 4444	4444 4444	3430 1344	0020 0000	0000 0000	0000 0000	n.d.
			60	4444 4444	4444 4444	4444 4444	4444 4444	4444 3430	0000 0000	0000 0000	0000 0000	n.d.

n.a. = not applicable
n.d. = not done

0 = no virus present; t = cytotoxic
1 to 4 = virus present (degree of CPE in 8 cell culture units) (wells of microtitre plates)

Table 5: Control of efficacy for suppression of test product activity (80.0 %) (2142)

Product	Interfering substance	Dilutions (log_{10})								
		1	2	3	4	5	6	7	8	9
Test product	PBS	tttt tttt	tttt tttt	4444 4424	0000 0100	0000 0000	0000 0000	0000 0000	0000 0000	n.d.
Test product	clean conditions	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Test product	dirty conditions	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

n.a. = not applicable
n.d. = not done

0 = no virus present; t = cytotoxic
1 to 4 = virus present (degree of CPE in 8 cell culture units) (wells of microtitre plates)

Table 6: Raw data (influenza A virus H1N1 swine) for cell sensitivity with columns (80.0 %) (2142)

Product	Interfering substance	Product dilution	Dilutions (log_{10})								
			1	2	3	4	5	6	7	8	9
PBS	PBS	-	4444 4444	4444 4444	4444 4444	4444 4444	4013 1330	0000 1000	0000 0000	0000 0000	n.d.
PBS	clean conditions	-	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
PBS	dirty conditions	-	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Test product	PBS	80.0%	4444 4444	4444 4444	4444 4444	4444 4444	4100 0000	0000 0000	0000 0000	0000 0000	n.d.
Test product	clean conditions	80.0%	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Test product	dirty conditions	80.0%	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.

n.a. = not applicable
n.d. = not done

0 = no virus present; t = cytotoxic
1 to 4 = virus present (degree of CPE in 8 cell culture units) (wells of microtitre plates)

Table 7: Summary of results with Germ Free 24 and influenza A virus H1N1 (swine)

Product	Con-centration	Interfering substance	Level of cytotoxicity	log_{10} $TCID_{50}$/ml after ....min									> 4 log_{10} reduction after ... min
				0	0.25	0.5	1.0	2.0	5.0	15.0	30.0	60.0	
product	80.0%	PBS	3.50	n.d.	n.d.	≤ 3.63	≤ 3.50	n.d.	n.d.	n.d.	n.d.	n.d.	≥ 1.0
product (col)	80.0%	PBS	≤ 1.50	n.d.	n.d.	≤ 2.38	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	≥ 0.5
product (col)	80.0%	PBS	≤ 1.50	n.d.	n.d.	n.d.	≤ 1.50	n.d.	n.d.	n.d.	n.d.	n.d.	1.0
product	10.0%	PBS	2.50	n.d.	n.d.	5.38	5.00	n.d.	n.d.	n.d.	n.d.	n.d.	> 1.0
form-aldehyde	0.7% (m/V)	PBS	4.50	n.d.	n.d.	n.d.	n.d.	n.d.	≤ 4.50	≤ 4.50	n.d.	n.d.	≥ 5.0
virus control	n.a.	PBS	n.a.	7.50/ 6.50	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	7.13/ 6.38	n.a.
virus control (col)	n.a.	PBS	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6.88/ 6.13	n.a.
sens. PBS (col)	n.a.	PBS	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	6.38	n.a.
sens. product (col)	n.a.	PBS	n.a.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	5.75	n.a.

n.a. = not applicable n.d. = not done col = columns