

Institute for Interlaboratory Studies

> Results of Proficiency Test OPP and other Preservatives in Textile December 2022

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1 INTRODUCTION

Many countries have adopted environmental standards and requirements restricting the use of harmful chemicals in the production of textiles and clothing. Laws and regulations impose some of these standards and requirements. In addition to mandatory environmental standards and requirements for textiles, some Eco-labelling schemes are imposing environmental requirements for textile products on a voluntary basis, e.g. Milieukeur (Netherlands), Bluesign® (Switzerland) and OEKO-TEX® Standard 100 (Switzerland).

Since 2004 the Institute for Interlaboratory Studies (iis) organizes a proficiency scheme for the determination of Ortho-Phenylphenol (OPP) in Textile every year, since 2019 this scheme is extended for other preservatives. During the annual proficiency test program 2022/2023 it was decided to continue the proficiency test of OPP and other Preservatives in Textile.

In this interlaboratory study 29 laboratories in 14 countries registered for participation, see appendix 4 for the number of participants per country. In this report the results of the OPP and other Preservatives in Textile proficiency test are presented and discussed. This report is also electronically available through the iis website www.iisnl.com.

2 SET UP

The Institute for Interlaboratory Studies (iis) in Spijkenisse, the Netherlands, was the organizer of this proficiency test (PT). Sample analyzes for fit-for-use and homogeneity testing were subcontracted to an ISO/IEC17025 accredited laboratory. It was decided to send one textile sample of approximately 3 grams labelled #22800. The participants were requested to report rounded and unrounded test results. The unrounded test results were preferably used for statistical evaluation.

2.1 QUALITY SYSTEM

The Institute for Interlaboratory Studies in Spijkenisse, the Netherlands, has implemented a quality system based on ISO/IEC17043:2010. This ensures strict adherence to protocols for sample preparation and statistical evaluation and 100% confidentiality of participant's data. Feedback from the participants on the reported data is encouraged and customer's satisfaction is measured on regular basis by sending out questionnaires.

2.2 PROTOCOL

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). This protocol is electronically available through the iis website www.iisnl.com, from the FAQ page.

2.3 CONFIDENTIALITY STATEMENT

All data presented in this report must be regarded as confidential and for use by the participating companies only. Disclosure of the information in this report is only allowed by means of the entire report. Use of the contents of this report for third parties is only allowed by written permission of the Institute for Interlaboratory Studies. Disclosure of the identity of one or more of the participating companies will be done only after receipt of a written agreement of the companies involved.

2.4 SAMPLES

A batch of orange cotton pieces was selected with a detectable level of OPP which was prepared by a third party. After homogenization 40 small plastics bags were filled with approximately 3 grams each and labelled #22800.

The homogeneity of the subsamples was checked by determination of Ortho-Phenylphenol (OPP) with an in house test method on 7 stratified randomly selected subsamples.

	OPP in mg/kg
sample #22800-1	47
sample #22800-2	55
sample #22800-3	53
sample #22800-4	51
sample #22800-5	56
sample #22800-6	57
sample #22800-7	52

Table 1: homogeneity test results of subsamples #22800

From the above test results the repeatability was calculated and compared with 0.3 times the reproducibility of the reference method in agreement with the procedure of ISO13528, Annex B2, in the next table.

	OPP in mg/kg
r (observed)	9.6
reference method	iis memo 1601
0.3 x R (reference method)	9.1

Table 2: evaluation of the repeatability of subsamples #22800

The calculated repeatability is in agreement with 0.3 times the reproducibility of the reference method. Therefore, homogeneity of the subsamples was assumed.

To each of the participating laboratories one textile sample labelled #22800 was sent on November 9, 2022.

2.5 ANALYZES

The participants were requested to determine Ortho-Phenylphenol (OPP), 2-(Thiocyanomethylthio)-Benzothiazole (TCMTB), 4-Chloro-3-Methylphenol (PCMC), 2-Octylisothiazol-3(2H)-one (OIT), Triclosan (TCS) and eventually other Preservatives detected.

To ensure homogeneity it was requested not to use less than 0.5 gram per determination. It was also requested to report if the laboratory was accredited for the determined components and to report some analytical details.

It was explicitly requested to treat the sample as if it was a routine sample and to report the test results using the indicated units on the report form and not to round the test results, but report as much significant figures as possible. It was also requested not to report 'less than' test results, which are above the detection limit, because such test results cannot be used for meaningful statistical evaluations.

To get comparable test results a detailed report form and a letter of instructions are prepared. On the report form the reporting units are given as well as the reference test methods (when applicable) that will be used during the evaluation. The detailed report form and the letter of instructions are both made available on the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The participating laboratories are also requested to confirm the sample receipt on this data entry portal. The letter of instructions can also be downloaded from the iis website www.iisnl.com.

3 RESULTS

During five weeks after sample dispatch, the test results of the individual laboratories were gathered via the data entry portal www.kpmd.co.uk/sgs-iis-cts/. The reported test results are tabulated per determination in appendices 1 and 2 of this report. The laboratories are presented by their code numbers.

Directly after the deadline, a reminder was sent to those laboratories that had not reported test results at that moment. Shortly after the deadline, the available test results were screened for suspect data. A test result was called suspect in case the Huber Elimination Rule (a robust outlier test) found it to be an outlier. The laboratories that produced these suspect data were asked to check the reported test results (no reanalyzes). Additional or corrected test results are used for data analysis and the original test results are placed under 'Remarks' in the result tables in appendices 1 and 2. Test results that came in after the deadline were not taken into account in this screening for suspect data and thus these participants were not requested for checks.

3.1 STATISTICS

The protocol followed in the organization of this proficiency test was the one as described for proficiency testing in the report 'iis Interlaboratory Studies: Protocol for the Organisation, Statistics and Evaluation' of June 2018 (iis-protocol, version 3.5). For the statistical evaluation the *unrounded* (when available) figures were used instead of the rounded test results. Test results reported as '<...' or '>...' were not used in the statistical evaluation.

First, the normality of the distribution of the various data sets per determination was checked by means of the Lilliefors-test, a variant of the Kolmogorov-Smirnov test and by the calculation of skewness and kurtosis. Evaluation of the three normality indicators in combination with the visual evaluation of the graphic Kernel density plot, lead to judgement of the normality being either 'unknown', 'OK', 'suspect' or 'not OK'. After removal of outliers, this check was repeated. If a data set does not have a normal distribution, the (results of the) statistical evaluation should be used with due care.

The assigned value is determined by consensus based on the test results of the group of participants after rejection of the statistical outliers and/or suspect data.

According to ISO13528 all (original received or corrected) results per determination were submitted to outlier tests. In the iis procedure for proficiency tests, outliers are detected prior to calculation of the mean, standard deviation and reproducibility. For small data sets, Dixon (up to 20 test results) or Grubbs (up to 40 test results) outlier tests can be used. For larger data sets (above 20 test results) Rosner's outlier test can be used. Outliers are marked by D(0.01) for the Dixon's test, by G(0.01) or DG(0.01) for the Grubbs' test and by R(0.01) for the Rosner's test. Stragglers are marked by D(0.05) for the Dixon's test, and by R(0.05) for the Rosner's test. Both outliers and stragglers were not included in the calculations of averages and standard deviations.

For each assigned value the uncertainty was determined in accordance with ISO13528. Subsequently the calculated uncertainty was evaluated against the respective requirement based on the target reproducibility in accordance with ISO13528. In this PT, the criterion of ISO13528, paragraph 9.2.1. was met for all evaluated tests, therefore, the uncertainty of all assigned values may be negligible and need not be included in the PT report.

Finally, the reproducibilities were calculated from the standard deviations by multiplying them with a factor of 2.8.

3.2 GRAPHICS

In order to visualize the data against the reproducibilities from literature, Gauss plots were made, using the sorted data for one determination (see appendix 1). On the Y-axis the reported test results are plotted. The corresponding laboratory numbers are on the X-axis. The straight horizontal line presents the consensus value (a trimmed mean). The four striped lines, parallel to the consensus value line, are the +3s, +2s, -2s and -3s target reproducibility limits of the selected reference test method. Outliers and other data, which were excluded from the calculations, are represented as a cross. Accepted data are represented as a triangle.

Furthermore, Kernel Density Graphs were made. This is a method for producing a smooth density approximation to a set of data that avoids some problems associated with histograms. Also, a normal Gauss curve (dotted line) was projected over the Kernel Density Graph (smooth line) for reference. The Gauss curve is calculated from the consensus value and the corresponding standard deviation.

3.3 Z-SCORES

To evaluate the performance of the participating laboratories the z-scores were calculated. As it was decided to evaluate the performance of the participants in this proficiency test (PT) against the literature requirements (derived from e.g. ISO or ASTM test methods), the z-scores were calculated using a target standard deviation. This results in an evaluation independent of the variation in this interlaboratory study.

The target standard deviation was calculated from the literature reproducibility by division with 2.8. In case no literature reproducibility was available, other target values were used, like Horwitz or an estimated reproducibility based on former iis proficiency tests.

When a laboratory did use a test method with a reproducibility that is significantly different from the reproducibility of the reference test method used in this report, it is strongly advised to recalculate the z-score, while using the reproducibility of the actual test method used, this in order to evaluate whether the reported test result is fit-for-use.

The z-scores were calculated according to:

```
z_{(target)} = (test result - average of PT) / target standard deviation
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The $z_{(target)}$ scores are listed in the test result tables in appendix 1.

Absolute values for z<2 are very common and absolute values for z>3 are very rare. Therefore, the usual interpretation of z-scores is as follows:

	z	< 1	good
1 <	z	< 2	satisfactory
2 <	z	< 3	questionable
3 <	z		unsatisfactory

4 EVALUATION

In this proficiency test no problems were encountered with the dispatch of the samples. Four participants reported test results after the final reporting date and three other participants did not report any test results. Not all participants were able to report all tests requested.

In total 26 laboratories reported 26 numerical test results. No statistical outliers were observed. In proficiency studies outlier percentages of 3% - 7.5% are quite normal.

Both data sets proved to have a normal Gaussian distribution.

4.1 EVALUATION PER COMPONENT

In this section the results are discussed per component. The test methods which were used by the various laboratories were taken into account for explaining the observed differences when possible and applicable. These test methods are also in the tables in appendix 1 together with the original data. The abbreviations, used in these tables, are explained in appendix 5.

Since 2019 test method EN17134 is available for the determination of OPP and Triclosan in textile. This test method describes an extraction with Acetonitrile using ultrasonic. Unfortunately, no precision data is mentioned in this method. Therefore, in this PT the test results will be evaluated against the target reproducibility as given in memo 1601. In iis memo 1601 an estimated iis target reproducibility based on iis PT data of OPP proficiency tests from 2004 until 2014 is determined.

Ortho-Phenylphenol (OPP): Based on the analytical details two groups of test results could be identified. It appeared that test results obtained from Acetonitrile and Alkaline extraction differ significantly (see paragraph 4.2 and 4.4 for more discussion). Therefore, it was decided to evaluate both groups separately. For the group using Acetonitrile as extraction solvent this determination was not problematic. No statistical outliers were observed. The calculated reproducibility is in agreement with the target reproducibility as derived from iis memo 1601.

For the group using KOH (followed by n-Hexane) as extraction solvent this determination may be problematic. No statistical outliers were observed but one test result was excluded. The calculated reproducibility after rejection of the suspect data is not in agreement with the target reproducibility as derived from iis memo 1601.

The participants agreed on a concentration near or below the limit of detection for all other components mentioned in paragraph 2.5. Therefore, no z-scores are calculated for these components. The reported test results are given in appendix 2.

4.2 PERFORMANCE EVALUATION FOR THE GROUP OF LABORATORIES

A comparison has been made between the reproducibility as declared by the reference test method and the reproducibility as found for the group of participating laboratories. The number of significant test results, the average, the calculated reproducibility (2.8 * standard deviation) and the target reproducibility derived from the reference method are presented in the next table.

Component	unit	n	average	2.8 * sd	R(target)
Ortho-Phenylphenol (OPP) ACN	mg/kg	16	12.2	8.5	8.6
Ortho-Phenylphenol (OPP) KOH	mg/kg	9	29.3	42.8	18.1

Table 3: reproducibilities of tests on sample #22800

Without further statistical calculations it can be concluded the participating laboratories have no difficulties with the determination of OPP when Acetonitrile is used as extraction solvent but have difficulties when KOH (followed by n-Hexane) is used as extraction solvent. See also the discussion in paragraphs 4.1, 4.4 and 5.

	December 2022	December 2021	December 2020	December 2019
Number of reporting laboratories	26	25	27	28
Number of test results	26	24	25	28
Number of statistical outliers	0	2	2	0
Percentage of statistical outliers	0%	8.3%	8.0%	0%

4.3 COMPARISON OF THE PROFICIENCY TEST OF DECEMBER 2022 WITH PREVIOUS PTS

Table 4: comparison with previous proficiency tests

iis PTs with OPP were combined with PCP Determination before 2019

In proficiency tests outlier percentages of 3% - 7.5% are quite normal.

The performance of the determinations of the proficiency test was compared to uncertainties observed in PTs over the years, expressed as relative standard deviation (RSD) of the PTS, see next table.

Component	December 2022	December 2021	December 2020	December 2019	December 2004-2018	Target *)
Ortho-Phenylphenol (OPP)	25-52%	35-36%	16-29%	21%	16-66%	20-34%

Table 5: development of the uncertainties over the years

*) derived from iis memo 1601 calculated at respectively 50-1.5 mg/kg

The uncertainties observed in this PT are comparable to the uncertainties observed in previous PTs.

4.4 EVALUATION OF THE ANALYTICAL DETAILS

For this PT some analytical details were requested which are listed in appendix 3. Based on the answers given by the participants the following can be summarized:

- About 75% of the participants mentioned that they are ISO/IEC17025 accredited to determine the reported component(s).
- Prior to analysis the samples were further cut by about 70% of the participants and about 30% used the sample as received.
- About 40% of the participants used a sample intake of 0.5 grams and about 60% used about 1 gram.
- Ultrasonic extraction was the most often reported technique to extract the components.
- About 60% of the participants used Acetonitrile and about 35% used KOH or KOH followed by n-Hexane as extraction solvent.

In this PT it is observed that the use of ACN or KOH as extraction solvent has a profound effect on the determination of OPP in the sample. An explanation could be that KOH breaks down the sample to release more OPP, while with ACN the free OPP is determined only. The effect of the choice of extraction solvent was also observed in the 2020 PT iis20A17 and in the 2021 PT iis21T09.Therefore, iis decided to evaluate both groups separately as it was done in 2020 and 2021 iis PTs.

The calculated reproducibility for Ortho-Phenylphenol (OPP) extracted with Acetonitrile is in agreement with the requirements of the target reproducibility, therefore no further separate statistical analysis has been performed.

5 DISCUSSION

All participants were able to detect OPP in sample #22800. However, the choice of the extraction solvent did influence the observed OPP amount and the decision about the acceptability or rejection of the sample.

For class 1 of OEKO-TEX® Standard 100 (see table 6) almost all participants would have rejected the sample, however three participants who used ACN and one participant who used KOH would have accepted the sample. For classes 2, 3 and 4 almost all participants would have accepted the sample, however five participants who used KOH would have rejected the sample.

Ecolabel	Class 1 Baby clothes in mg/kg	Class 2 Clothes direct skin contact in mg/kg	Class 3 Clothes, no direct contact in mg/kg	Class 4 Decoration material in mg/kg
Ortho-Phenylphenol (OPP)	10	25	25	25

Table 6: OEKO-TEX® Standard 100

In this PT, the average of the homogeneity test results are not in line with the average (consensus value) from the PT results. There are several reasons for this. First, the goal of the homogeneity testing is very different from the goal of the evaluation of the reported PT results. In order to prove the homogeneity of the PT samples, a test method is selected with a high precision (smallest variation). The accuracy (trueness) of the test method is less relevant.

Secondly, the homogeneity testing is done by one laboratory only. The test results of this (ISO/IEC 17025 accredited) laboratory will have a bias (systematic deviation) depending on the test method used. The desire to detect small variations between the PT samples leads to the use of a sensitive test method with high precision, which may be a test method with significant bias.

Also, each test result reported by the laboratories that participate in the PT will have a bias. However, some will have a positive bias and others a negative bias. These different biases compensate each other in the PT average (consensus value). Therefore, the PT consensus value may deviate from the average of the homogeneity test. At the same time the accuracy of the PT consensus value is more reliable than the accuracy of the average of the results of the homogeneity test.

6 CONCLUSION

In this proficiency test Ortho-Phenylphenol (OPP) and other Preservatives in Textile were determined. In this PT it is observed that the choice of extraction solvent has an effect on the determination of OPP in the sample. The choice of extraction solvent should be dependent on use of the test results. This means that for the determination of released OPP the ACN extraction is needed and the determination of total OPP the use of KOH extraction.

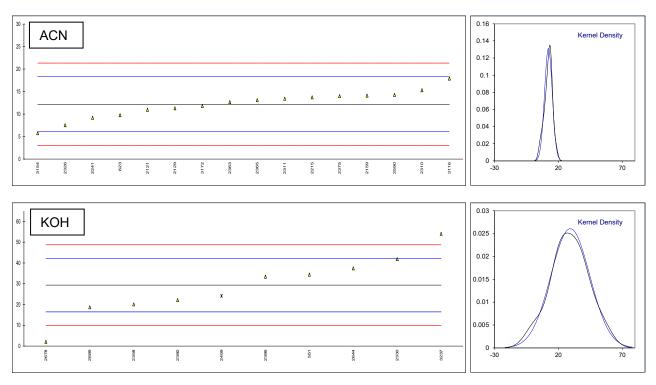
Each participating laboratory will have to evaluate its performance in this study and decide about any corrective actions if necessary. Therefore, participation on a regular basis in this scheme could be helpful to improve the performance and thus increase of the quality of the analytical results.

APPENDIX 1

Determination of Ortho-Phenylphenol (OPP) on sample #22800; results in mg/kg

lab	method	ACN	mark	z(targ)	КОН	mark	z(targ)
551	§64 LFGB B82.02.8				34.3731		0.78
623	ISO13365	9.75		-0.79			
2121	EN17134	10.949		-0.40			
2129		11.28		-0.29			
2159	In house	14.10		0.63			
2215	EN17134	13.72		0.51			
2241	EN17134	9.176		-0.98			
2265							
2293							
2310	ISO13365	15.3		1.02			
2311	EN17134	13.417		0.41			
2326	EN17134	7.53		-1.52			
2330	§64 LFGB B82.02.8Mod.				41.89		1.94
2358					20.096		-1.43
2363	ISO13365-1	12.68		0.17			
2365	EN17134	13.1		0.30			
2375	EN17134	14		0.60			
2380	LFGB B82.02.8Mod.				22.139		-1.12
2386	In house				33.44		0.63
	ISO13365-1				24.13	ex	-0.81
2590	EN17134	14.269		0.69			
	UNI11057				37.41		1.25
2678					2.05		-4.23
	In house				18.6776		-1.65
2977							
3116	In house/ISO13365	17.9		1.87			
	EN17134	5.77		-2.09			
3172		11.804		-0.12			
3237	In house				54.07		3.83
	normality	ОК			ок		
	n	16			9		
	outliers	0			0 + 1ex		
	mean (n)	12.172			29.350		
	st.dev. (n)	3.0432	RSD = 25%		15.2974	RSD = 52%	
	R(calc.)	8.521			42.833		
	st.dev.(iis-memo 1601)	3.0570			6.4598		
	R(iis-memo 1601)	8.560			18.087		

Lab 2459 reported to have used Methanol as extraction solvent, therefore the test result was excluded for statistical evaluation



APPENDIX 2 Other reported test results

- TCMTB = 2-(Thiocyanomethylthio)-Benzothiazole
- PCMC = 4-Chloro-3-Methylphenol
- OIT = 2-Octylisothiazol-3(2H)-one
- TCS = Triclosan

Determination individual and other Preservatives on sample #22800; in mg/kg

lab	ТСМТВ	PCMC	OIT	TCS	Other Preservatives
551		0.1997			
623	Not Detected				
2121					
2129	<10	<10	<10	<10	<10
2159	not determined	not determined	not determined	not determined	not applicable
2215	Not determined	Not detected	Not detected	Not detected	Not determined
2241				<2.0	
2265					
2293					
2310	not detected				
2311	Not Detected				
2326				ND	
2330	Not applicable				
2358	not detected				
2363	<1.0	<1.0	<1.0	<1.0	not analyzed
2365	<1.0	<1.0	<1.0	<1.0	
2375					
2380					
2386	< 5	< 5	< 5	< 10	
2459	ND	ND	ND	ND	ND
2590					
2644	not analyzed	not analyzed	not analyzed	not determined	not analyzed
2678				ND	
2689					
2977					
3116					
3154	not detected				
3172	< 5	< 5	< 5		
3237					

APPENDIX 3 Analytical details

lab	ISO17025	Sample	Sample intake	Extraction	Extraction
	accredited	preparation	(grams)	technique	solvent
551	Yes	Further cut	1g	Ultrasonic	KOH followed by n-Hexane
623	Yes	Further cut	1	Ultrasonic	Acetonitrile
2121	Yes	Used as received	1g	Ultrasonic	Acetonitrile
2129	Yes	Used as received	0,5	Ultrasonic	Acetonitrile
2159	Yes	Further cut	1 gram	Ultrasonic	Acetonitrile
2215	No	Further cut	1g	Ultrasonic	Acetonitrile
2241	Yes	Further cut	0.5g	Ultrasonic	Acetonitrile
2265					
2293					
2310	Yes	Further cut	1	Ultrasonic	Acetonitrile
2311	Yes	Further cut	0.5	Ultrasonic	Acetonitrile
2326	Yes (OPP), No (Triclosan)	Further cut	1 gm	Ultrasonic	Acetonitrile
2330	No	Further cut	0.50 g	Ultrasonic	KOH followed by n-Hexane
2358	No	Used as received	1.0	Alkaline Digestion	KOH followed by n-Hexane
2363	Yes	Used as received	1.0g	Ultrasonic	Acetonitrile
2365	Yes	Further cut	1.0g	Ultrasonic	Acetonitrile
2375	Yes	Further cut	0.5 gram	Ultrasonic	Acetonitrile
2380	Yes	Further cut	1.0 g	Alkaline Digestion	KOH followed by n-Hexane
2386	Yes	Further cut	0.501 g	Ultrasonic	OPP: KOH, other preservatives: Methanol
2459	No	Further cut	1.0 gram	Ultrasonic	Methanol
2590	Yes	Used as received	1g	Ultrasonic	Acetonitrile
2644	No	Further cut	0.5	Mechanical Shaking	KOH followed by n-Hexane
2678	No	Used as received	1 gram	Ultrasonic	KOH followed by n-Hexane
2689	Yes	Further cut	0.5g	Ultrasonic	KOH followed by n-Hexane
2977					
3116	No	Used as received	1 gram	Ultrasonic	Acetonitrile
3154	Yes	Used as received	0,5 g	Ultrasonic	Acetonitrile
3172	Yes	Further cut		Ultrasonic	Acetonitrile
3237	Yes	Further cut	0,5	Mechanical Shaking	KOH followed by n-Hexane

APPENDIX 4

Number of participants per country

1 lab in BANGLADESH

- 1 lab in BRAZIL
- 1 lab in CAMBODIA
- 1 lab in FRANCE
- 4 labs in GERMANY
- 1 lab in GUATEMALA
- 2 labs in HONG KONG
- 2 labs in INDIA
- 1 lab in INDONESIA
- 4 labs in ITALY
- 5 labs in P.R. of CHINA
- 2 labs in PAKISTAN
- 1 lab in TUNISIA
- 3 labs in TURKEY

APPENDIX 5

Abbreviations

С	= final test result after checking of first reported suspect test result
D(0.01)	= outlier in Dixon's outlier test
D(0.05)	= straggler in Dixon's outlier test
G(0.01)	= outlier in Grubbs' outlier test
G(0.05)	= straggler in Grubbs' outlier test
DG(0.01)	= outlier in Double Grubbs' outlier test
DG(0.05)	= straggler in Double Grubbs' outlier test
R(0.01)	= outlier in Rosner's outlier test
R(0.05)	= straggler in Rosner's outlier test
E	= calculation difference between reported test result and result calculated by iis
W	= test result withdrawn on request of participant
ex	= test result excluded from statistical evaluation
n.a.	= not applicable
n.e.	= not evaluated
n.d.	= not detected
fr.	= first reported
f+?	= possibly a false positive test result?
f-?	= possibly a false negative test result?

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