

Area della Ricerca CNR, Via Moruzzi, 1 - Loc. S. Cataldo – 56124 PISA Tel. 050-315.2552/2550 - Fax 050-315.2555 - e-mail <u>cempini@icas.pi.cnr.it</u>

EXECUTIVE SUMMARY

INVESTIGATION OF SUITABILITY OF PROOFING SEALERS TO THE CONTACT WITH FOODSTUFF

Introduction

This report presents the results obtained by the Migration Test of chemical substances, among those declared by the producing Company forming the liquid proofing sealers for stone surfaces, in consequence of the realization of covering layers by the same sealers on different materials.

Two different samples of proofing liquids, manufactured by the Company GENERAL, have been received from CGS s.a.s. of Pisa:

- IMPERSTON
- IMPERSTON NATURA

Both the samples of liquids have been delivered into their original containers bearing the label of the manufacturing company with the instrucions for use and sealed by screw cap.

From the moment they have been received, the samples have been kept into their respective containers and kept at controlled temperature (20°C).

Meaning and aim of the research:

- Checking the presence of chemical substances migrated from the proofing covering applied to different kind of materials; application made according to the instructions separately given by the manufacturing company and sent to us by CGS s.a.s.
- Evaluation if such eventual presence, when in contact with foodstuff, can be cause of risk to the health of the final users.

Criteria adopted for the research:

Taking into cosideration the concerned Regulations and what specified at the previous point, the research has been made through the following phases:

- Arrangement of samples (parallelepipeds of approximate thickness of 1 cm. and flat surface of 50 cm²) of each of the material below specified: Marble Granite
 - Terracotta
- On each kind of material, and according to specification of the manufacturing Company, application of a layer of proofing sealer onto the flat surface of each sample by using one of the two products.
- Insertion and conservation of the samples in contact with the simulating liquid into a closed glass chamber, at controlled temperature, for a proper period.
- Repetition of the test on the same sealed samples with simulating liquid exactly alike to the simulating liquid previously used.
- Use of analytical techniques for the survey of the eventual migration.
- Comparison of the results.



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General information

In compliance with the Ministerial Decree dated March 21, 1973 and subsequent modifications and integrations (addendum A), the suitability of a product in contact with foodstuff is fixed by executing analytical tests, named *Migration Tests*; the tests put the product in contact (*attack*) with a liquid that simulates the extractive power of a kind of foodstuff (*simulating liquid*).

The target of the test is the analysis of the simulating liquid which results are usually expressed in terms of:

- Total Migration (mg/dm²)
- Specific Migration (mg/L or ppm) of a fixed chemical family.

Here below is the list of the simulating liquids expected for the transfer tests, together with the foodstuff categories they represent:

SIMULATING LIQUID	REPRESENTED FOODSTUFF CATEGORY
A: Distilled Water	Neutral Aqueous Foodstuff
B: Solution at 3% (w/w) of Acetic Acid	Acid Aqueous Foodstuff
C: Solution at 10% (w/w) of Ethanol	Neutral Alcoholic Foodstuff
D: Rectified Olive oil or Sunflower oil or the ⁽¹⁾ " <i>test means</i> ": Isooctane, Ethanol 99%, MPPO ⁽²⁾	Oily and Fatty Foodstuff

(1) The Ministerial Decree dated July 22, 1998 provides for "substitute tests" and "alternative tests" to the use of simulating liquids of the fatty substances. The *substitute tests* use *test means* in the "conventional conditions of the substitute test; they can be choosen when, because of technical reasons concerning the method of analysis, it is not possible to effect the Migration Test by using rectified olive oil or rectified sunflower oil.

⁽²⁾ MPPO: Modified PolyPhenyl Oxide

For each test are expected three subsequent attacks and the analysis of the liquid coming from the third attack.

Duration and temperatures of the migration tests are correlated to the destination of use of the tested material. Among the conditions provided by the Ministerial Decree dated March 21, 1973 and subsequent Decrees, for the products tested during this reasearch the conditions selected are those provided by the section concerning the procedures.

Reference limits

TOTAL MIGRATION

Total Migration Limit (TML) allowed (by EU) for a migration test: 10 mg/dm². Total Migration Limit (TML) allowed by Italian Regulations (M.D. dtd March 21, 1971): 8 mg/dm².

SPECIFIC MIGRATION

As the Regolations don't provide for limit values for the under specified chemical families, we think useful to remember some correlated limit values provided for Italian and International Regulations.

FLUORIDES:

- Maximum concentration allowed for drinking waters (Directive 80/778 EU): 1,5 ppm
- Maximum concentration allowed for drinking waters (National Primary Drinking Water Regulations, EPA): 4 ppm
- Maximum Fluoride extracted (U.S. FDA *CFR*): 0,46 mg/dm².
- Maximum Fluoride extracted, calculated as concentration contained into the volume of the simulating liquid in contact with the product (U.S. FDA *CFR*): 5 ppm
- •



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CHLORIDES:

• Maximum concentration allowed for drinking waters (Directive 80/778 EU): 200 ppm

SULPHATES:

• Maximum concentration allowed for drinking waters (Directive 80/778 EU): 250 ppm

HEAVY METALS:

Maximum concentration allowed for drinking waters:

Element	Directive 80/778 EU	NPDWR ⁽³⁾ , EPA	
	ppm	ppm	
Fe	0,3		
Cu	0,05	1,3	
Zn	3	5 (NSDWR) (3)	
Al		0,2 (NSDWR) (3)	
As	0,05	0,05	
Cd	0,005	0,005	
Pb	0,05	0,015	
Se	0,01	0,05	
Cr	0,05	0,1	
Hg	0,001	0,002	
Ba	0,1		

NPDWRs: National Primary Drinking Water Regulations NSDWRs: National Secondary Drinking Water Regulations

Methods of analysis (4)

Ponderal Tests (Ministerial Decree March 21, 1973): Total Migration

Ion Choromatography (EPA /600/R-93/100, ASTM 11.01, 11.02): Anions

ICP-MS (EPA/600/R-94/111): Metals

IR: Characterisation, TOC (5)

NMR ¹H, ¹⁹F, ¹³C: Characterisation and Fluorine

⁽⁴⁾ See Addendum B

⁽⁵⁾ For completness, in the case of the simulating A, measurements have been effected of TOC (Total Organic Carbon) and TC (Total Carbon = Total Organic Carbon + Inorganic Carbon), even if the Regulations don't provide for limits of specific migration for such method.



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Equipment

Infrared Spectroscopy

Perkin-Elmer FTIR 1725X spectrophotometer, single ray, windows for KBr in 4000-400 cm⁻¹ area. The spectra have been processed by using the softwares IRplus, Lotus 1-2-3, and Microsoft Excel.

Spectroscopy of Nuclear Magnetic Resonance

Varian Gemini 200 BB device. The spectra have been processed by using the softwares Acorn NMR Nuts, Mestre-C, and ACD SpecViewer.

Ion Chromatography

Dionex Series 4500i fitted with Pulsed Electrochemical Detector, and connected to the Laboratory Computing Integrator Perkin-Elmer LCI-100.

Chromatographic conditions:

- Coloumn: Dionex IonPac AS9-SC Analytical Coloumn
- Guard: AG9-SC
- Eluent: 1.8 mM Na₂CO₃ / 1.7 mM Na₂CO₃
- Regenerant: 25 mM H₂SO₄
- Regenerant flow rate: 5.0 mL/min



Standard used:

Chromatogram concerning the Standard DIONEX; according the eluation sequence:

- Fluorides 2 ppm
- Chlorides 3 ppm
- Nitrates 10 ppm
- Phosphates 15 ppm
- Sulphates 15 ppm

ICP-MS

Varian Ultra-Mass-700 ICP-Mass Spectrometer Sample Preparation System: Varian SPS-5 Internal Standard:

- In 25 ppb
- Cs 25 ppb
- Y 25ppb

TOC 2000 Apparatus



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Procedures

The migration tests - marked as <u>Test 1</u>, <u>Test 2</u>, <u>Test 3</u> for the simulating liquids A, B, C, and marked as <u>Test A</u>, <u>Test B</u>, <u>Test C</u> for the simulating liquids D and the *test means* – were consisting of:

- <u>Test 1</u>: "Short contact" for 24 hours at temperature of 20°C (3 attacks, analysis of the liquid of the third attack)
- <u>Test 2</u>: "Short contact" for 30 minutes at temperature of 25°C (3 attacks, analysis of the liquid of the third attack)
- <u>Test 3</u>: "Short contact" for 2 hours at temperature of 40°C (3 attacks, analysis of the liquid of the third attack)
- <u>Test A:</u> "Short contact" for 2 hours at temperature of 40°C (3 attacks, analysis of the liquid of the third attack)
- <u>Test B:</u> "Short contact" for 24 hours at temperature of 20°C (3 attacks, analysis of the liquid of the third attack)

Ponderal Test (Ministerial Decree March 21, 1073)

The simulating liquid coming from the Migration Test (1,2,3,A or B) is collected, the solvent is evaporated, the residuum is dessicated (105°C) till to constant weight, and cooled for 30 minutes into a dessicator and finally weighed.

The Total Migration, expressed as mg/dm², is calculated as e/S where:

e = residuum mass in mg.

S = Area of material put in contact with the simulating liquid espressed in dm^2 .

ANALYTIC DATA

All the tests have been effected on samples of marble, terracotta and granite treated with the products under examination according to the specification of the manufacturing Company, let dried 96 hours after the treatment, and then polished by a clean cotton cloth.

After this phase the samples have been put in contact with the simulating liquid, in full compliance with the ratio area/volume provided for the Ministerial Decree March 21, 1973.

The samples have been collected together in initialled series according to the treatment made and the simulating liquid used, by the *legenda* "Serie XN" where:

X = initial of the simulating liquid used (A,B,C,D)

N = kind of treatment:

- 1 = a treatement with IMPERSTON NATURA
- 2 = a treatment with IMPERSTON

Each serie bears the indication of the tests effected (by the *legenda*: Test 1, Test 2, Test 3, Test A, Test B)

In every single serie the samples are numbered and initialled with G (granite), M (marble), C (cotto). Each test has been effected by using at least 3 samples subjected to equal treatment.

No significant difference of migration between the samples 1, 2, or 3 has been found; the analytic results shown are a weighed average of the results of the different tests.



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Simulating liquid A

Samples of Granite, Marble have been subjected to migration tests with distilled water, purified by Milli-Q (*Millipore*) system.

SERIE A1: Test 1, 2 and 3

- Sample G
- Sample M

PONDERAL TEST:

The average total migration shown by the samples of the Serie A1 is: A1: G: 2,0 mg/dm² A1: M: 0,1 mg/dm²

IR ANALYSIS:

The samples G, M of the serie A1 have shown to the infrared only the bands of absorption because of the water Milli-Q, that means absence of migration from the material under examination.

CHROMATOGRAPHIC ANALYSIS:

The analysis made by ionic chromatography shown, for all the samples, presence of chloride, that anyway is fully kept within the limits.

The quantitative results are shown in the table a1

Table a1: quantified anions per IC on some samples of the serie A1		
sample # Cl		
G1	8.76 ppm	
G2	8.89 ppm	
M1	6.22 ppm	
M2	6.65 ppm	



picture 1: chomatogram of the sample G1



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ICP-MS ANALYSIS:

The analysis made have shown absence of significant concentrations of metal cations into the simulating liquid (**table a2**). The elements not listed in the table have shown concentrations lower than 1 ppb.

Table a2 : metal cations into simulating liquids of the serie A1 (average value)		
Element Concentration (ppb)		
Na 23	95	
Mg 24	24	
AI 27	90	
Zn 66	6	
Sr 88	26	

TOC ANALYSIS:

The TOC average value of the samples of the serie A1 are shown in the table a3

Table a3: TOC average value on some samples of the serie A1			
Sample	TOC (ppm) Total Crganic Carbon	TC (ppm) Total Carbon	
M1	1.359	2.132	
M2	1.440	2.127	
G1	1.474	2.330	
G2	1.501	2.480	
G3	1.507	2.267	

SERIE A2: Test 1, 2 and 3

- Samples G.
- Samples M.

IR ANALYSIS :

because

The infrared spectrum of all the samples shows an evident bow around 1712 cm⁻¹ due to the strectching of the group MeCOO (acetate), that is absent in the white (water Milli-Q).

CHROMATOGRAPHIC ANALYSIS:

By the ionic chromatography the presente oc Acetate has been found in all the samples treated with IMPERSTON: by this technique the signal due to the acetate can interfere (like in our case) when



determining the Fluoride of very close times of retention.



picture 2: Chromatogram of two samples (coming from attendion machine and granite sigh traited with 144 FISTON) diluted 1:100 showing the interform preak of these relations 0.50-315.2555 - e-mail compini@icas.pi.cnr.it

The repetition of the analysis after the preparation of the serie A2 with new samples of marble and granite, with following washing of the same samples with water Milli-Q, shown these results:

PONDERAL TEST:

The average total migration coming from of the series A2 is: A2: G: 7.2 mg/dm² A2: M: 4.0 mg/dm^2

IR ANALYSIS:

Only one of the new samples (M4) evidenced the bow due to the acetate, whilst all the other have not evidenced differences with the spectrum of the white (water Milli-Q).

CHROMATOGRAPHIC ANALYSIS:

The chromatographic analysis effected on the new serie have shown the presence of Chlorides, Fluorides and Sulphates in four samples. In the samples evidencing migration of Fluoride the quantity of Fluorides exceeds the quantity allowed by EU Regulations for drinking waters, whilst comply with the parameters fixed by EPA for this kind of waters.

The results are shown in the table a4.

Table a4: Anions, quantified per IC, on some samples of the serie A2				
sample # F CI SO4				
M1		16.23 ppm	10.03 ppm	
M2		11.64 ppm	2.32 ppm	
G1	1.79 ppm	5.72 ppm		
G2	2.98 ppm	8.26 ppm		





picture 3: chromatogram, sample G1

picture 4: chromatogram, sample G2

ICP-MS ANALYSIS:

The analysis made have shown absence of significant concentrations of metal cations into the simulating liquid (**table a5**). The elements not listed in the table have shown concentrations lower than 1 ppb.

Table a5 : metal cations into simulating liquids of the serie A2 (average value)		
Element Concentration (ppb)		
Na 23	542	
Mg 24	56	
Zn 66	5	
Sr 88	15	
Ba 137	2	



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TOC ANALYSIS:

The TOC average value of the samples of the serie A2 are shown in the table a6

Table a6: TOC average value on some samples of the serie A2			
Sample	TOC (ppm) Total Crganic Carbon	TC (ppm) Total Carbon	
M1	1.570	2.310	
M2	1.652	2.406	
G1	1.929	2.800	
G2	1.935	2.794	

After the results evidenced by the first analysis of samples treated with IMPERSTON, the series examinated afterwards have been submitted to the following treatment before the migration test:

- The surface has been washed three times with water Milli-Q
- The surface has been got dry by the air

The same samples of the series A1 and A2 have been examinated again after an interval of 10 days, and put again in contact with the simulating liquid and the analysis has been effected again; no difference of results has been found.

Simulating liquid B

Samples of Granite and Marble have been submitted to migration test by means of a solution 3% (w/w) of Acetic Acid and water Milli-Q.

SERIE B1: Test 1, 2 and 3

- Samples G.
- Samples M.

SERIE B2: Test 1, 2 and 3

- Samples G.
- Samples M.

PONDERAL TEST

The average total migration shown by the samples of the Serie B1 is:

- B1: G: 1.1 mg/dm^2
- B1: M: 0.9 mg/dm^2

The average total migration shown by tha samples of the Serie B2 is:

- B2: G: 6.5 mg/dm^2
- B2: M: 3.3 mg/dm²



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IR ANALYSIS

The samples of Marble and Granite of the Series B1, B2, treated with IMPERSTON don't show significant migration revealable into the liquid coming from the third attack and they essentially show the same infrared spectrum of the white (simulant solution B). Nevertheless it must be remarked that an eventual migration of acetates it should not be revealable because covered by the acetates obviously present into the simulating liquid.

Because of the tried interference of the acetates on the IC analysis, the analysis by the lonic Chromatography has not been possible:

<u>NMF ¹⁹F ANALYSIS</u> It is not releavable the presence of Fluorine.

Simulating liquid C

Samples of Granite and Marble have been submitted to migration test by means of a solution 10% (w/w) of Ethyl Alcohol and water Milli-Q.

SERIE C1: Test 1, 2 and 3

- Samples G.
- Samples M.

SERIE C2: Test 1, 2 and 3

- Samples G.
- Samples M.

PONDERAL TEST

The average total migration shown by the samples of the Serie B1 is:

C1: G: 0.9 mg/dm^2

C1: M: 0.4 mg/dm²

The average total migration shown by the samples of the Serie B2 is:

- C2: G: 4.9 mg/dm^2
- C2: M: 2.1 mg/dm²

IR ANALYSIS

The infrared spectra of the samples of the Series C1 and C2 don't show difference compared with the spectrum concerning the simulating liquid, evidencing absence of migration.

<u>NMF ¹⁹F ANALYSIS</u> It is not releavable the presence of Fluorine.



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Simulating liquid D

Surfaces of Granite, Terracotta and Marble have been submitted to migration tests by rectified Olive Oil and by Ethanol 95% (Isooctane, Ethanol 95%, and MPPO⁽⁶⁾ are called *test means* suitable as "substitute tests" of migration tests effected with simulating D: Law Decree 338/98, - see note # (1) page 2)

⁽⁶⁾ Modified PolyPhenyl Oxide

SERIE D1: Test A and B

- Samples G.
- Samples M.
- Samples C.

SERIE D2: Test A and B

- Samples G.
- Samples M.
- Samples C.

PONDERAL TEST:

The average total migration shown by the samples of the Series D1 is:

- D1: G: 0.7 mg/dm^2
- D1: M: 0.5 mg/dm²
- D1: C: 1.1 mg/dm^2

The average total migration shown by the samples of the Series D2 is:

- D2: G: 4.6 mg/dm^2
- D2: M: 3.4 mg/dm²
- D2: C: 7.1 mg/dm^2

IR ANALYSIS:

The infrared spectra effected on the samples of the Series D1 and D2 show the same absotption bands shown by the simulating liquid or by the pure *test mean*.

NMF ¹⁹F ANALYSIS

The spectra NMF¹⁹F shown absence of quantity of Fluorine releavable both in the samples of simulating liquid and the *test means* used for the attack.



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CONCLUSION

After the examination of the results obtained during the tests effected on surfaces covered with a layer of proofing product, it is possible conclude that:

- The product IMPERSTON NATURA shown an extremely reduced release, even when put in contact with alcoholic or acid substances; on the whole it demonstrates to be inert and resistant to the action of the simulating liquids.
- The product IMPERSTON, after washing with water, shown a limited migration of Fluorides, Chlorides, Sulphates and Acetates. The Fluorides exceed the limits fixed for the drinking waters by CEE (EU) but they are within the limits fixed by EPA; the other components don't exceed the limits fixed by CEE (EU) Regulations.

It is suggested a re-examination of the product for reducing the migration.

In the light of the Regulations in force, no contraindication seems to be expressed for the use of the products IMPERSTON and IMPERSTON NATURA, as per samples sent by us by GENERAL company, in superficial claddings of Marble, Granite and Terracotta. So it is certified that the above mentioned products can be in contact with foodstuff and derivatives for short times at room temperature.

Pisa, February 26, 2001

The Director

Prof G. Raspi





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ADDENDUM A

Modifications to D.M. (Ministerial Decree) 21 Marzo 1973

D.M. 03/08/74 D.M. 19/11/74 D.M. 27/03/75 D.M. 13/09/75 D.M. 18/06/79 D.M. 26/03/80 D.M. 02/12/80 D.M. 25/06/81 D.M. 23/08/81 n. 777 D.M. 20/10/82 D.M. 18/02/84 D.M. 04/04/85 D.M. 02/06/82 D.M. 07/08/87 n. 395 D.M. 01/06/88 n. 243 D.M. 18/01/91 n. 90 D.M. 30/10/91 n. 408 D.M. 25/01/92 n. 108 D.M. 26/04/93 n. 220 D.M. 15/07/93 D.M. 20/09/93 D.M. 03/06/94 n. 511 D.M. 01/07/94 n. 566 D.M. 28/10/94 n. 735 D.M. 08/02/95 D.M. 24/02/95 n. 156 D.M. 05/0595 D.M. 13/07/95 n. 405 D.M. 24/09/96 n. 572 D.M. 06/02/97 n. 91 D.M. 22/07/98 D.M. 04/08/99 D.M. 17/12/99





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ADDENDUM B

Methods of analysis approved by EPA and ASTM

Contaminant	Methodology	EPA	ASTM
Alkalinity	Titrimetric Electrometri titration		D1067-92B
Antimony	ICP-Mass Spectrometry Hydride-Atomic Absorption Atomic Absorption; Platform Atomic Absorption; Furnace	200.8 200.9	D3697-92
Arsenic	Inductively Coupled Plasma ICP-Mass Spectrometry Atomic Absorption; Platform Atomic Absorption; Furnace Hydride-Atomic Absorption	200.7 200.8 200.9	
Asbestos	Transmission-Electron-Microscopy Transmission-Electron-Microscopy	100.1 100.2	
Barium	ICP-Mass Spectrometry Hydride-Atomic Absorption Atomic Absorption; Platform Atomic Absorption; Furnace	200.7 200.8	
Berillium	ICP-Mass Spectrometry Hydride-Atomic Absorption Atomic Absorption; Platform Atomic Absorption; Furnace	200.7 200.8	
Cadmium ^₄	ICP-Mass Spectrometry Hydride-Atomic Absorption Atomic Absorption; Platform Atomic Absorption; Furnace	200.7 200.8 200.9	
Calcium	EDTA titrimetric Atomic Absorption; Direct Inductively-coupled plasma	200.7	D511-93A D511-93B



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Chromium	ICP-Mass Spectrometry Hydride-Atomic Absorption Atomic Absorption; Platform Atomic Absorption; Furnace	200.7 200.8 200.9	
Copper	Atomic Absorption; Furnace Atomic Absorption; Direct Inductively Coupled Plasma ICP-Mass Spectroscopy Atomic Absorption; Platform	200.7 200.8 200.9	D1688-95C D1688-95A
Conductivity	Conductance		D1125-95A
Cyanide	Preliminary Distillation Step Spectrophotometric, Amenable Spectrophotometric, Manual Semi-automated Selective Electrode	335.4	D2036-91A D2036-91B D2036-91A
Fluoride	Ion Chomatrography Preliminary Distillation Step SPADNS Colorimetric Manual Electrode Automated Electrode Automated Alizarin	300.0	D4327-91 D1179-93B
Lead	Atomic Absorption; Furnace ICP-Mass spectrometry Atomic Absorption; Platform Differential Pulse Anodic Stripping Voltammetry	200.8 200.9	D3559-95D
Magnesium	Atomic Absorption ICP Complexation Titrimetric Methods	200.7	D511-93B D511-93B
Mercury	Manual, Cold Vapor Automated, Cold Vapor ICP-Mass Spectrometry	245.1 245.2 200.8	D3223-91



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Nickel	Inductively Coupled Plasma ICP-Mass Spectrometry Atomic Absorption; Plaform Atomic Absorption; Direct Atomic Absorption; Furnace	200.7 200.8 200.9	
Nitrate	Ion Chromatography Automated CD Reduction Ion Selective Electrode Manual Cadmium Reduction	300.0 353.2	D4327-91 D3867-90A D3867-90B
Nitrite	Ion Chromatography Automated CD Reduction Manual Cadmium Reduction Spectrophotometric	300.0 353.2	D4327-91 D3867-90A D3867-90B
Ortho-phosphate unfiltered, no digestion or hydrolysis	Colorimetric: - Automated, Ascorbic Acid - Ascobic Acid, single reagent Phosphomolybdate - Automated-segmented flow - Automated discrete	365.1	D515-88A
	Ion Chromatography	300.0	D 4327-91
рН	Electrometric	150.1 150.2	D1293-95
Selenium	Hydride-Atomic Absorption ICP-Mass Spectrometry Atomic Absorption; Plaform Atomic Absorption; Furnace	200.8 200.9	D3859-93A
Silica	Colorimetric: Automated-seg. flow Molybdosilicate Heteropoly blue Automated method for molybdate-reactive silica Inductively-coupled Plasma	200.7	D859-95



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Sodium	Inductively-coupled Plasma Atomic Absorption; Direct	200.7	
Temperature	Thermometric		
Thallium	ICP-Mass Spectrometry Atomic Absorption; Platform	200.8 200.9	