



# Standards

## SPEX Standards for the Superfund Contract Laboratory Program

SPEX Industries introduces a complete series of multi-element standards and blanks designed for use in the U.S. Environmental Protection Agency's Contract Laboratory Program, CLP. These off-the-shelf solutions provide everything you need for:

- Instrument Calibrations
- Calibration Verifications
- Interference Checks
- Calibration Blanks
- Sample Spikes

Every standard is prepared under the unique **SPEX Triple-Checked Quality Assurance Program**. This program guarantees the purity and concentration of your standards for one full year. Only SPEX plasma-grade standards are checked three ways:

- DC arc trace metal analysis of starting materials
- Classical "wet" assay of major elements
- ICP check of the final solution

For over 30 years SPEX has meant Quality, Reliability and Convenience to the inorganic spectroscopist.... At SPEX spectroscopy is our business.

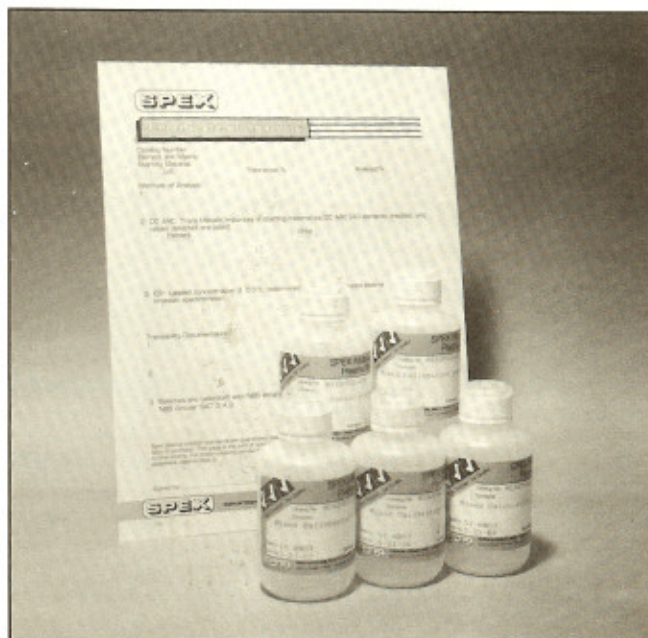
**QUALITY:** We provide **QUALITY** solution standards for ICP, DCP, and AA. Trace impurities can interfere with the element being analyzed, giving incorrect results. SPEX not only uses the highest purity materials available, we analyze and report trace impurities in our standards as well as the major elements.

**RELIABILITY:** Spectroscopists **RELY** on SPEX. All standards are triple-checked in our own laboratories, starting with trace impurity analysis via DC arc through "wet" assay for major constituent and finally ICP analysis. Analysis certificates are provided to our customers for all of our ICP-grade standards showing the results of all three analyses with traceability documentation. SPEX guarantees the accuracy of your standard to  $\pm 0.5\%$  for one year.

**CONVENIENCE:** We make it **CONVENIENT**. Save time, money and man-hours. Our standards are designed to fit your needs. From the initial calibration solution to the post digestion spike all you require is a straightforward dilution and you're ready to go. Or for your special applications we can design custom standards with the same high quality and the same guarantees.

Our order department and chemical sales staff are waiting to assist you. Let us put over 30 years of knowledge to work for your laboratory. If you have a standardization or purity problem, call us for advice, recommendations and reliable SPEX products to solve that problem.

Request the full-line **SPEX Catalog of Standards and Compounds** for every application of inorganic spectroscopy.



The following standards and solutions have been designed by SPEX Industries for use in conjunction with the U.S. Environmental Protection Agency's Contract Laboratory Program, CLP. They are to be used in conjunction with the Statement of Work, SOW.7/87, Rev. 12/87. Dilutions that determine the final concentrations of the various elements are suggested for each solution. The analyst may, however, choose to modify these dilution factors since the absolute concentration, to some extent, may be determined by the sample to be analyzed and the instrumentation used.

Please refer to the **SPEX Catalog of Standards for Plasma Emission Spectroscopy** for additional single- and multi-element standards that can also be used in these studies. SPEX offers a complete line of off-the-shelf and custom standards designed for a variety of environmental and industrial applications.

The U.S. EPA SOW.7/87 gives specific procedures for the methods of analysis, elements to be analyzed for, and concentration levels. Standards are specified not only by the elements present and their relative concentrations, but also the order and frequency of running standards, blanks and samples. Details of these specifications may be found in the EPA SOW.7/87 in the following sections:

- Exhibit C, Inorganic Target Analyte List, TAL
- Exhibit D, Analytical Methods
- Exhibit E, QA/QC Requirements



## Instrument Calibration

The series of solutions below may be used separately or mixed together for calibration of the instrument and preparation of the analytical curve. When mixed these solutions will yield a standard containing all the elements in the Target Analyte List. Instruments must be calibrated daily, every 24 hours or each time the instrument is set up. Calibration standards must be prepared fresh for each analysis and discarded after use. A dilution of 100-fold is suggested for ICAL-2, ICAL-3, and ICAL-4, and a dilution of 10-fold for ICAL-1. Antimony and mercury can be diluted as required.

### Instrument Calibration Standard 1

ICAL-1	100ml
Matrix 5% HNO <sub>3</sub>	
Ca	5000ug/ml
Mg	5000ug/ml
K	5000ug/ml
Na	5000ug/ml

### Instrument Calibration Standard 2

ICAL-2	100ml
Matrix 5% HNO <sub>3</sub>	
Ag	100ug/ml
Cr	100ug/ml
Mn	150ug/ml
Ni	400ug/ml
Zn	200ug/ml

### Instrument Calibration Standard 3

ICAL-3	100ml
Matrix 5% HNO <sub>3</sub>	
Al	2000ug/ml
Ba	2000ug/ml
Be	50ug/ml
Co	500ug/ml
Cu	250ug/ml
Fe	1000ug/ml
V	500ug/ml

### Instrument Calibration Standard 4

ICAL-4	100ml
Matrix 5% HNO <sub>3</sub>	
As	100ug/ml
Cd	50ug/ml
Pb	50ug/ml
Se	50ug/ml
Ti	100ug/ml

### Instrument Calibration Standard 5

ICAL-5	100ml
Matrix 2% HNO <sub>3</sub>	
Sb	600ug/ml

### Instrument Calibration Standard 6

ICAL-6	100ml
Matrix 2% HNO <sub>3</sub>	
Hg	100ug/ml

## Initial Calibration Verification

After the instrument is calibrated, the calibration must be verified, at each wavelength to be used for analysis, with an independent standard. SPEX verification standard, ICV-1, contains all the elements on the TAL in one solution. You need only select those wavelengths/elements for verification that are to be subsequently analyzed. A dilution of 10-fold is recommended prior to use.

### Initial Calibration Verification Standard

ICV-1	500ml
Matrix 5% HNO <sub>3</sub>	

Al	200ug/ml
Sb	60ug/ml
As	10ug/ml
Ba	200ug/ml
Be	5ug/ml
Cd	5ug/ml
Ca	5000ug/ml
Cr	10ug/ml

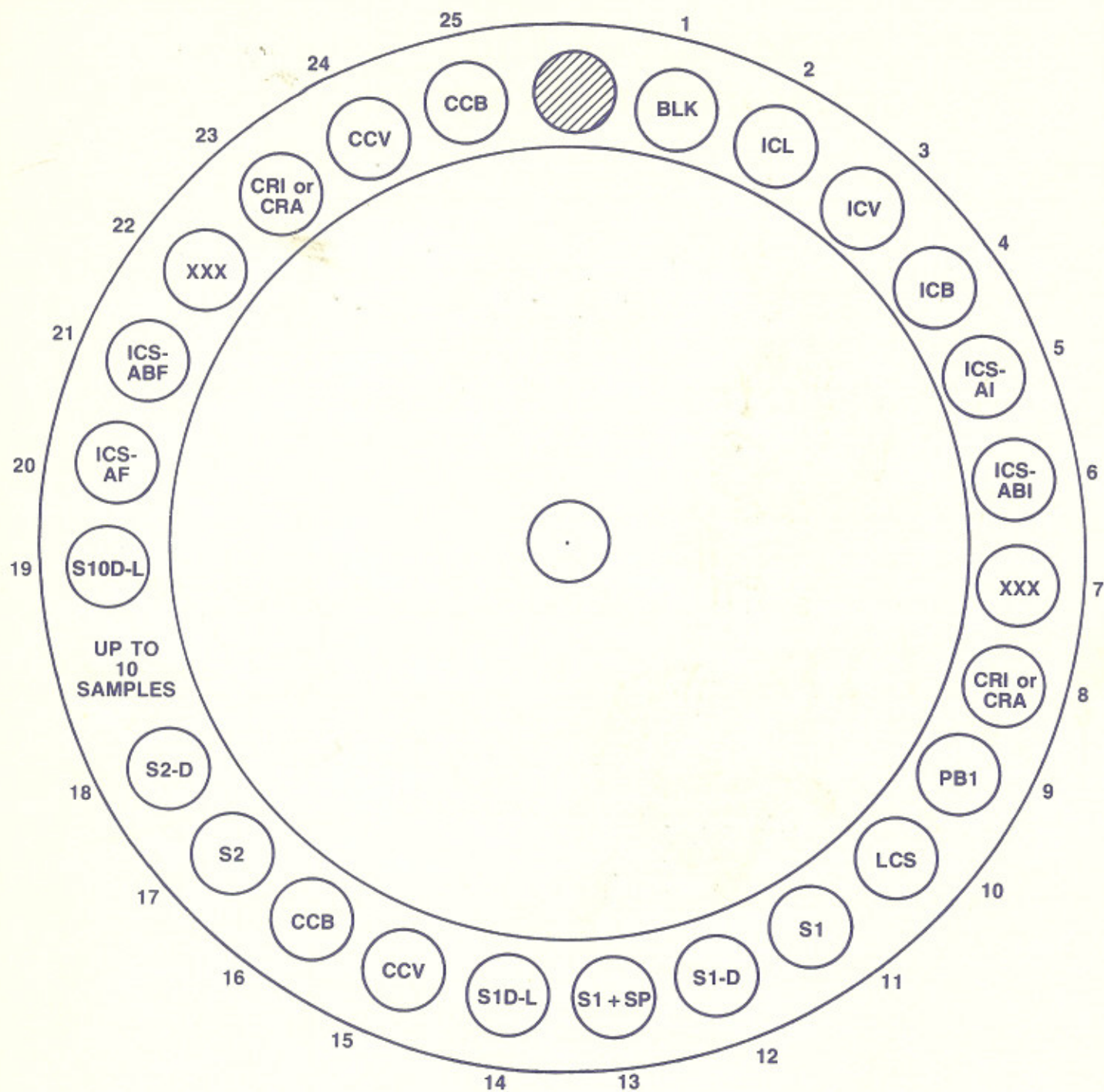
Co	50ug/ml
Cu	25ug/ml
Fe	100ug/ml
Pb	5ug/ml
Mg	5000ug/ml
Mn	15ug/ml
Ni	40ug/ml
K	5000ug/ml

Se	5ug/ml
Ag	10ug/ml
Na	5000ug/ml
Ti	10ug/ml
V	50ug/ml
Zn	20ug/ml

## Contract Required Detection Limits, CRDL

A standard must be run at two times the Contract Required Detection Limits, CRDL, or at two times the Instrument Detection Limits, IDL, whichever is greater. This standardization must be performed at the start and the end of each sample analysis or at least twice in an 8 hour shift. All elements to be analyzed must be run except Al, Ba, Ca, Fe, Mg, Na, and K.

The CRDL-1 standard contains all the required elements on the TAL, in their appropriate concentration ratios. CRDL-1 should be diluted by a factor of 1000 prior to use in the "two times CRDL" run for ICP analysis. For analysis by atomic absorption, CRDL-1 should be diluted by a factor of 2000 prior to use in the "one times CRDL" run. The elements arsenic and lead should be run at two times the Instrument Detection Limit, IDL. The concentrations of these elements may be adjusted with the corresponding SPEX single-element standards for arsenic, PLAS2-2X and lead, PLPB2-2X. This standard may also be used as an IDL standard or alternatively the Instrument Calibration Standards can be mixed and diluted accordingly.



Typical set-up for standards, Samples, and blanks to be run for CLP analysis.



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## SPEX Standards For The Contract Laboratory Program

Following is a list of samples, standards, and blanks in a possible running sequence as suggested by the Contract Laboratory Program protocols. Also listed are the SPEX standards and solutions to be used in preparing the blanks, standards and spikes.

1. BLK - Blank: SPEX PLBLK's
2. ICL - Initial calibration solution: mixture of SPEX ICAL's
3. ICV - Initial calibration verification: SPEX ICV-1
4. ICB - Initial calibration blank (not digested): SPEX PLBLK's
5. ICS-AI - Initial interferences, A: SPEX INT-A1 and INT-A2
6. ICS-ABI - Initial interferences and analytes AB: SPEX INT-A1, INT-A2, INT-B1, INT-B2
7. XXX - "Throw-away" sample; used to clear torch of high levels of interferences: SPEX PLBLK's
8. CRI (ICP) or CRA (AA) - 2 x Contract required detection limits: SPEX CRDL-1
9. PB1 - Preparation blank: Digested water or soil blank
10. LCS - Laboratory control sample (digested): SPEX ICV-1
11. S1 - Sample #1
12. S1-D - Sample #1 duplicate
13. S1 + SP - Sample #1 with spike: SPEX SPIKE-1
14. S1D-L - Sample #1 diluted five fold
15. CCV - Continuing calibration verification (50% ICV): SPEX ICV-1
16. CCB - Continuing calibration blank: SPEX PLBLK's (If results of CCV and CCB are within limits, proceed to next sample, if not stop run)
17. S2 - Sample #2
18. S2-D - Sample #2 duplicate
- Up to 10 samples may be run as long as CCV and CCB tests are within accepted limits.
19. S10D-L - Sample #10 diluted five fold
20. ICS-AF - Final interferences, A: SPEX INT-A1 and INT-A2
21. ICS-ABF - Final interferences, and analytes, AB: SPEX INT-A1, INT-A2, INT-B1, INT-B2
22. XXX - "Throw-away" sample; used to clean torch of high levels of interferences: SPEX PLBLK's
23. CRI (ICP) or CRA (AA) 2 x Contract required detection limits: SPEX CRDL-1
24. CCV - Continuing calibration verification (50% ICV): SPEX ICV-1
25. CCB - Continuing calibration blank: SPEX PLBLK's

### Price List Standards for The Contract Laboratory Program

Catalog Number	Volume	Description	Price Each
ICAL-1	100 ML	Instrument Calibration Standard 1	100.00
ICAL-2	100 ML	Instrument Calibration Standard 2	69.00
ICAL-3	100 ML	Instrument Calibration Standard 3	85.00
ICAL-4	100 ML	Instrument Calibration Standard 4	60.00
ICAL-5	100 ML	Instrument Calibration Standard 5	29.00
ICAL-6	100 ML	Instrument Calibration Standard 6	29.00
ICV-1	500 ML	Initial Calibration Verification	395.00
CRDL-1	100 ML	Contract Required Detection Limits	120.00
INT-A1	500 ML	Interferences A	200.00
INT-B1	100 ML	Analytes B	100.00
INT-A2	500 ML	Alternate Interferences A	170.00
INT-B2	100 ML	Alternate Analytes B	105.00
SPIKE-1	100 ML	Spike Sample Standard	150.00
CLP-SET		Set above consists of one each of the above solutions, 13 in all	1100.00
PLBLK-HNO3	500 ML	5% Nitric Acid Blank	29.00
PLBLK-HCL	500 ML	5% Hydrochloric Acid Blank	29.00
PLBLK- H2O	500 ML	ASTM Type I Water	29.00

Prices are subject to change without notice



**Contract Required Detection Limits Standard**

CRDL-1 ..... 100ml  
 Matrix 5% HNO<sub>3</sub>

Sb ..... 120ug/ml  
 As ..... 20ug/ml  
 Be ..... 10ug/ml  
 Cd ..... 10ug/ml  
 Cr ..... 20ug/ml

Co ..... 100ug/ml  
 Cu ..... 50ug/ml  
 Pb ..... 6ug/ml  
 Mn ..... 30ug/ml  
 Ni ..... 80ug/ml

Se ..... 10ug/ml  
 Ag ..... 20ug/ml  
 Tl ..... 20ug/ml  
 V ..... 100ug/ml  
 Zn ..... 40ug/ml

## Calibration Blanks and Matrix Blanks

All SPEX blank solutions are prepared with the same high-purity acids and ASTM Type I water as that used in our plasma-grade standards. SPEX matrix blanks can be used to dilute standards or can be run directly as blanks to establish base lines. The calibration blank is prepared by mixing one part nitric acid blank with four parts hydrochloric acid blank.

**Nitric Acid Blank**

PLBLK-HNO<sub>3</sub> ..... 500ml  
 Matrix 5% HNO<sub>3</sub> in ASTM Type I Water

**Hydrochloric Acid Blank**

PLBLK-HCL ..... 500ml  
 Matrix 5% HCl in ASTM Type I Water

**Water Blank**

PLBLK-H<sub>2</sub>O ..... 500ml  
 Matrix ASTM Type I Water, 18 megohm

## Interference Checks

Interelement and background correction factors must be verified at the beginning and the end of each analysis run or at least twice in an 8 hour shift. Two solutions are required for the interference check: the interferents alone, solution A, and the combination of interferents and analytes, solution AB. Solution A is prepared by diluting INT-A1 by a factor of 10. Solution AB is prepared by diluting (together) solution A by a factor of 10 and solution B by a factor of 100; for example diluting 10ml of solution A plus 1.0ml of solution B to 100ml with calibration blank.

**Interferents A**

INT-A1 ..... 500ml  
 Matrix 5% HNO<sub>3</sub>

Al ..... 5000ug/ml  
 Ca ..... 5000ug/ml  
 Fe ..... 2000ug/ml  
 Mg ..... 5000ug/ml

**Analytes B**

INT-B1 ..... 100ml  
 Matrix 5% HNO<sub>3</sub>

Ag ..... 100ug/ml  
 Ba ..... 50ug/ml  
 Be ..... 50ug/ml  
 Cd ..... 100ug/ml  
 Co ..... 50ug/ml  
 Cr ..... 50ug/ml

Cu ..... 50ug/ml  
 Mn ..... 50ug/ml  
 Ni ..... 100ug/ml  
 Pb ..... 100ug/ml  
 V ..... 50ug/ml  
 Zn ..... 100ug/ml

In addition to the four interferents contained in INT-A1, the EPA SOW.7/87 lists six more elements that can interfere with analysis of the elements on the TAL. SPEX also provides a set of alternate interference samples that may be used in conjunction with the standards above. Together they enable the analyst to check and correct for all the interferences listed in SOW.7/87.

Again two solutions are required. The first solution, A\*, is prepared by combining and diluting INT-A1 and INT-A2 by a factor of ten: 10ml of INT-A1 plus 10ml of INT-A2 diluted to 100ml with calibration blank. The second solution, AB\*, requires both interferent standards diluted by a factor of 10 and both the analytes diluted by a factor of 100: 10ml of INT-A1, plus 10ml of INT-A2, plus 1.0ml of INT-B1, plus 1.0ml of INT-B2, diluted to 100ml with calibration blank.

**Alternate Interferents A**

INT-A2 ..... 500ml  
 Matrix 5% HNO<sub>3</sub>

Cr ..... 1000ug/ml  
 Cu ..... 1000ug/ml  
 Mn ..... 1000ug/ml  
 Ni ..... 1000ug/ml  
 Ti ..... 1000ug/ml  
 V ..... 1000ug/ml

**Alternate Analytes B**

INT-B2 ..... 100ml  
 Matrix 5% HNO<sub>3</sub>

Al ..... 100ug/ml  
 As ..... 100ug/ml  
 B ..... 100ug/ml  
 Ca ..... 10ug/ml  
 Fe ..... 10ug/ml  
 Mg ..... 10ug/ml

Mo ..... 100ug/ml  
 Na ..... 100ug/ml  
 Sb ..... 100ug/ml  
 Se ..... 100ug/ml  
 Si ..... 10ug/ml  
 Tl ..... 100ug/ml

## Spike Sample Analysis

In the spike sample analysis a spike containing the required elements, in their respective required amounts, is added to the sample prior to addition of any reagents, digestion, distillation, etc. Information is then provided on the effects of the sample matrix and the entire methodology. The SPEX spike standard, SPIKE-1, provides all the analytes required for the ICP and the AA spike. Add 1.0ml of SPIKE-1 to aqueous samples and 2.0ml of SPIKE-1 to solid samples prior to digestion.

### Spike Sample Standard

SPIKE-1 ..... 100ml  
Matrix 5% HNO<sub>3</sub>

Al ..... 200ug/ml  
Sb ..... 50ug/ml  
As ..... 200ug/ml  
Ba ..... 200ug/ml  
Be ..... 5ug/ml  
Cd ..... 5ug/ml

Cr ..... 20ug/ml  
Co ..... 50ug/ml  
Cu ..... 25ug/ml  
Fe ..... 100ug/ml  
Pb ..... 50ug/ml  
Mn ..... 50ug/ml

Ni ..... 50ug/ml  
Se ..... 200ug/ml  
Ag ..... 5ug/ml  
Tl ..... 200ug/ml  
V ..... 50ug/ml  
Zn ..... 50ug/ml

## Furnace AA Calibration and Spiking

Furnace AA requires calibration standards and spikes containing only six elements: Sb, As, Cd, Pb, Se, and Tl. These solutions are easily prepared by diluting **Instrument Calibration Standard 4, ICAL-4**, and **Instrument Calibration Standard 5, ICAL-5**, to the required concentrations.

## Ordering Information

**Telephone:** 1-201-549-7144  
1-800-LAB-SPEX  
(1-800-522-7739)

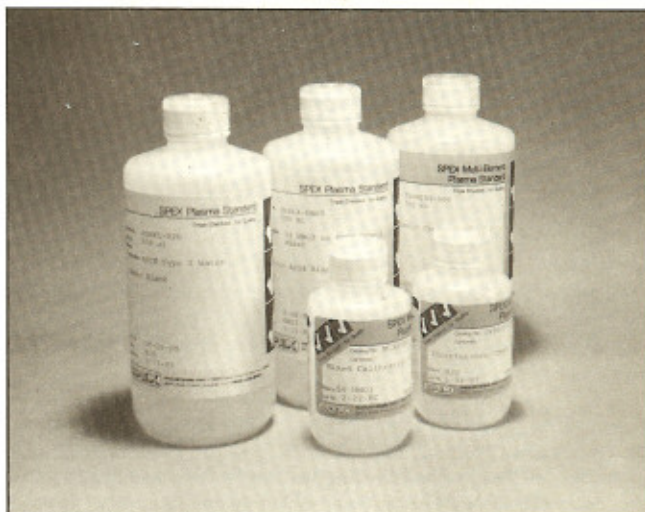
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**Precautions:**

SPEX products are not for any cosmetic, drug or household application. Our acceptance of a purchase order is with the assumption that only qualified individuals, trained and familiar with procedures suitable to the products ordered, will handle them. On our clients must rest the entire burden of safe storage, handling, and application of all products ordered from this catalog.



# SPEX

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