

NICKEL, VANADIUM, IRON, LEAD, COPPER AND SODIUM IN GAS OILS BY AAS

UOP Method 848-84

SCOPE

This method is for determining nickel, vanadium, iron, lead and copper in gas oils by graphite furnace electrothermal Atomic Absorption Spectrophotometry (GFAAS) at levels below 2000 ng/mL (approximating mass-ppb). For determination of higher levels, see UOP 800. Sodium content, expressed as $\mu\text{g/g}$ (mass-ppm), is determined directly by flame AAS. The lower limit of detection for all metals, including sodium, ranges from about 2 to 20 ppb depending on the element sought.

OUTLINE OF METHOD

The gas oil is wet ashed with concentrated sulfuric acid and the resultant char is burned off in a muffle furnace. The remaining inorganic residue (ash) is dissolved in hydrochloric and nitric acids. The sample solution is diluted to the proper reading concentrations and analyzed by graphite furnace atomic absorption. Peak heights of samples are compared to the peak heights of standards in a mixed acid matrix. Concentrations of the metals sought are then calculated for the sample.

Sodium is determined by direct aspiration into the flame of the AAS instrument after dilution with xylene and alcohols. A standard addition technique is used.

APPARATUS

Atomic absorption spectrophotometer, having a direct readout (or recorder output) proportional to absorbance units, flame, graphite furnace accessory and background corrector accessory. The Perkin-Elmer 603 with Model 2200 HGA is one example. An automatic sampler is also helpful but is not necessary. If an automatic sampling system is not used, then an Eppendorff automatic pipeter, 20- μL capacity, with plastic disposable tips, must be used, Sargent-Welch Scientific Co., Cat. Nos. S-69721-DS and S-69722-AA, respectively, or equivalent.

Boiling burner, triple slot, 100-mm, preferred over single slot burner

Balance, readability, 0.1 mg

Balance, top loading, readability, 0.01 g

Beakers, 250-mL, heat resistant type, Vycor or quartz glass

IT IS THE USER'S RESPONSIBILITY TO ESTABLISH APPROPRIATE PRECAUTIONARY PRACTICES AND TO DETERMINE THE APPLICABILITY OF REGULATORY LIMITATIONS PRIOR TO USE. EFFECTIVE HEALTH AND SAFETY PRACTICES ARE TO BE FOLLOWED WHEN UTILIZING THIS PROCEDURE. FAILURE TO UTILIZE THIS PROCEDURE IN THE MANNER PRESCRIBED HEREIN CAN BE HAZARDOUS. MATERIAL SAFETY DATA SHEETS (MSDS) OR EXPERIMENTAL MATERIAL SAFETY DATA SHEETS (EMSDS) FOR ALL OF THE MATERIALS USED IN THIS PROCEDURE SHOULD BE REVIEWED FOR SELECTION OF THE APPROPRIATE PERSONAL PROTECTION EQUIPMENT (PPE).

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Bottles, narrow mouth, round, with molded screw cap, 120-mL capacity, Sargent-Welch Scientific, Cat. No. S-8240-C, or equivalent

Bottles, plastic, Nalgene type, 30-mL and one-liter

Clamps, utility, V jaw, Sargent-Welch Scientific, Cat. No. S-19160, or equivalent, 3 required

Column, chromatographic, with fritted disk, 20-mm ID, 400-mm length, complete with 500-mL funnel, Corning Glass Works, Cat. No. 32152-20, or equivalent

Cylinder, graduated, glass, 10-mL

Dropper, one-piece, disposable, Nalgene No. 6219-0068, Fisher Scientific, Cat. No. 13-711-10, or equivalent

Flasks, volumetric, 10-, 25-, 50-, 100-, 200-mL and one-liter, several of each required

Funnel, plastic disposable, 55-mm diameter, 25-mm stem length, 69-mm height

Furnace, muffle, electric, large enough to hold three to four 250-mL beakers

Gloves, rubber, lightweight industrial, Playtex, Sargent-Welch Scientific, Cat. No. S-40268, or equivalent

Hot plate, electric, a continuous and variable surface temperature of 15-200 C is required, Corning PC-100, or equivalent

Hollow cathode lamps, (HCL), iron, nickel, vanadium, lead, copper and sodium

Oven, large enough to hold 3 to 4 sample bottles

Pipet, Mohr, one-milliliter capacity, Sargent-Welch Scientific, Cat. No. S-69555-C, or equivalent

Pipets, 1-, 2-, 5-, and 10-mL, several of each required

Ring stand, sufficient height to support column and separatory funnel

Stirring rods, Vycor, heavy duty, 150-mm length

Tongs, beaker

Watch glasses, 90-mm diameter, borosilicate glass

REAGENTS AND MATERIALS

All reagents shall conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit use without lessening the accuracy of the determination. References to water mean distilled or deionized water.

Acetylene, cylinder compressed gas, welders grade, recommend size 1A (300 cu ft), equipped with a C₂H₂ regulator, i.e., Perkin-Elmer, Cat. No. 303-0106, or equivalent

Air, compressed, filtered and dried, minimum of 415 kPa (60 psig) to AAS instrument

Aluminum nitrate, 98% minimum purity

Argon, 99.995% purity, cylinder, Size No. 1A, 8.21 m³ (290 ft³)

1-Butanol, 99% minimum purity

Copper, metal wire or powder, 99.999% purity, SPEX Industries, or equivalent

Copper stock solution, 1000mg/L. Weigh 1.000 g $\pm$ 0.1 mg of primary standard copper metal powder or wire into a 250-mL beaker. Add 100 mL of water and slowly add 30 mL of concentrated nitric acid. Cover with a watch glass and heat gently until dissolution is complete. Transfer quantitatively to a one-liter volumetric flask, allow to cool and dilute to the mark with water and mix.

Copper solution, 10 mg/L. Pipet 10 mL of the 1000-mg/L standard into a one-liter volumetric flask. Dilute to the mark with water and mix.

Copper solution, 1 mg/L. Pipet 10 mL of the 10-mg/L standard into a 100-mL volumetric flask. Dilute to the mark with water and mix.

Hydrochloric acid, concentrated, ULTREX Ultrapure Reagent, J. T. Baker, Cat. No. 4800, or equivalent

Ion exchange resin, macroreticular, Amberlyst, Grade 15, Rohm and Haas, or equivalent

Iron, metal sponge or wire, 99.999% purity, SPEX Industries, or equivalent

Iron, stock solution, 1000 mg/L. Weigh 1.000 g $\pm$ 0.1 mg of primary standard iron sponge or wire into a 250-mL beaker. Add 100 mL of water and 10 mL of concentrated hydrochloric acid. Cover the beaker with a watch glass and warm gently until the wire or sponge is completely dissolved. Cool the solution and transfer it quantitatively to a one-liter volumetric flask. Dilute to the mark with water and mix.

Iron solution, 10 mg/L. Pipet 10 mL of the 1000-mg/L standard into a one-liter volumetric flask and dilute to the mark with water and mix.

Iron solution, 1 mg/L. Pipet 10 mL of the 10-mg/L standard into a 100-mL volumetric flask and dilute to the mark with water and mix.

Lead nitrate, 99.999% purity, SPEX Industries, or equivalent

Lead stock solution, 1000 mg/L. Weigh 1.5985 g $\pm$ 0.1 mg of lead nitrate into a beaker. Dissolve in 1% (v/v) nitric acid in water and transfer quantitatively into a one-liter volumetric flask. Dilute to the mark with the same nitric acid and mix.

Lead solution, 10 mg/L standard. Pipet 10 mL of the 1000-mg/L standard into a one-liter volumetric flask, dilute to the mark with water and mix.

Lead solution, 1 mg/L standard. Pipet 10 mL of the 10-mg/L standard into a 100-mL volumetric flask, dilute to the mark with water and mix.

Methanol, 99% minimum purity

Nickel, metal sponge, 99.999% purity, SPEX Industries, or equivalent

Nickel stock solution, 1000 mg/L. Weigh 1.000 g $\pm$ 0.1 mg pure metal into a 250-mL beaker, wet with approximately 100 mL of water and slowly add 30 mL of nitric acid. Cover with a watch glass and heat gently until dissolution is complete. Transfer quantitatively to a one-liter volumetric flask; dilute to the mark with water and mix.

Nickel solution, 10 mg/L. Pipet 10 mL of the 1000-mg/L standard into a one-liter volumetric flask. Dilute to the mark with water and mix.

Nickel solution, 1-mg/L standard. Pipet 10 mL of the 10-mg/L stock standard solution into a 100-mL volumetric flask and dilute to the mark with water and mix.

Nitric acid, concentrated, ULTREX Ultrapure Reagent, J. T. Baker, Cat. No. 4801, or equivalent

Sodium octoate, organic standard, Angstrom, Inc., or equivalent. The material is supplied as an accurately known oil solution at approximately the 4 mass-% sodium level.

Sodium stock solution, 100 mg/kg. Weigh the calculated amount of sodium octoate organic standard to ± 0.1 mg into a tared 120-mL bottle. Add xylene to obtain a total mass of 100 g.

Sodium solution, 0.5 $\mu\text{g/g}$. Weigh 0.5 g ± 0.1 mg of the 100-mg/kg stock solution into a tared 120-mL bottle. Add xylene to obtain a total mass of 100 g.

Sulfuric acid, concentrated, 0.2 mass-ppm maximum iron content, E. I. DuPont de Nemours and Co., electronic grade, available from Sargent-Welch Scientific, or equivalent

Vanadium, metal pieces or turnings, 99.999% purity, SPEX Industries, or equivalent

Vanadium stock solution, 1000 mg/L. Weigh 1.000 g ± 0.1 mg of standard metal into a 250-mL beaker and cautiously add 10 mL of concentrated nitric acid; cover with a watch glass and gently heat until dissolution is complete. Add 100 mL of H_2O , heat to a boil, and transfer quantitatively into a one-liter volumetric flask. Dilute to the mark with water and mix.

Vanadium solution, 10 mg/L. Pipet 10 mL of the 1000-mg/L standard into a one-liter volumetric flask; dilute to the mark with water and mix.

Vanadium solution, 1 mg/L. Pipet 10 mL of the 10-mg/L standard into a 100-mL volumetric flask; dilute to the mark with water and mix.

Xylene, 99.9% minimum purity.

PROCEDURE

Metals Other Than Sodium

Wet Ash Blank Processing

Prepare and process a wet ash blank at the same time samples are being processed, in the following manner. Into a 250-mL Vycor beaker transfer 10 mL of concentrated sulfuric acid. Evaporate to dryness on a hot plate and allow the beaker to cool. Continue to process this blank in the same manner as instructed under *Sample Processing*, starting at the second paragraph “To the cool beaker...” and continue to the end of that paragraph.

Sample Processing

Warm the sample in an oven until it is fluid and waxes are melted. To obtain a representative sample, shake the sample in its container for at least one minute. Using a top loading balance weigh 20 ± 0.01 g of oil into a 250-mL Vycor beaker. Cover the beaker with a watch glass and add 10 mL of concentrated sulfuric acid (see Note 1). Place a Vycor stirring rod in the beaker and heat it on a hot plate at 90-150 C in a hood or well ventilated area. The oil will react with the sulfuric acid producing varying amounts of a coke-like char (voluminous white fumes will indicate reaction). If the sample foams excessively, temporarily remove it from the heat and replace it when foaming subsides. Continue to heat until the reaction is almost complete. Break up the char with the stirring rod. Continue to heat for about 2 hours and then raise the hot plate temperature to 175-200 C. To help prevent refluxing of liquids in the beaker and aid in the removal of SO_3 fumes, set the watch glass so that it only partially covers the beaker. When the remainder of unreacted oil has charred and excess sulfuric acid has evaporated, raise the hot plate surface temperature to 340-370 C.

Stir occasionally with the Vycor rod to break up lumps. When fuming stops and the sample appears dry, place the covered beaker containing the Vycor rod in a muffle furnace at 540 ± 25 C (see Note 2). After 2 hours, partially uncover the beaker by supporting the watch glass on the rod. Continue to heat until the char is reduced to ash (preferably overnight). Replace the watch glass to cover the beaker. Remove the beaker from the muffle furnace and allow it to cool.

To the cool beaker, covered with the watch glass and containing the Vycor stirring rod, and one milliliter of concentrated hydrochloric acid and one milliliter of concentrated nitric acid. Place on a hot plate at about 90 C and heat until the ash is dissolved, being careful not to boil away acids. Transfer quantitatively into a 50-mL volumetric flask. Allow the solution to cool and then bring to the mark with water. For any additional sample dilutions designate **T** as the aliquot from the 50 mL volumetric flask and **F** the final volume to which the aliquot is diluted (see *CALCULATIONS*).

Analysis

Conditions for setting the instrument are found in the following Table 1.

Table 1 Instrument Parameters					
Element	Fe	Ni	V	Cu	Pb
Source			HCL		
Wavelength	248.3 nm	232.0 nm	318.4 nm	324.7 nm	283.3 nm
Slit setting	0.2 nm	0.2 nm	0.7 nm	0.7 nm	0.7 nm
Background correction			yes		
Purge gas			argon		
Argon flow rate			40 mL/minute		
Sample size			20- μ L		
Graphite tube type			pyro-coated		
Recorded speed			10 mm/minute		
Expansion (suggested)			2X		

Furnace Conditions					
Element	Fe	Ni	V	Cu	Pb
Maximum power	no	yes	yes	no	no
Dry temperature			150 C		
Dry time			25 sec		
Char temperature	1200 C	1000 C	1500 C	900 C	500 C
Char time			25 sec		
Atomization temperature	2500 C	2400 C	2700 C	2700 C	2400 C
Atomization time			8 sec		

Standard Concentration

The following are suggested standard concentrations, which may be modified depending on the specific operations. Always use a minimum of 2 standards and a blank.

Table 2
Concentrations in ng/mL

Element	Fe	Ni	V	Cu	Pb
Blank, % HNO ₃	0.2	0.2	0.2	0.2	0.2
			+ Al		
1st Standard	10	25	100	10	25
2nd Standard	20	50	200	20	50

Iron Analysis

Set the instrument parameters and furnace conditions for iron. Make fresh working standards daily preferably just before instrument use.

To prepare a 0.1 µg/mL Fe solution, pipet 10 mL of 1-mg/L solution into a 100-mL volumetric flask, dilute to the mark with water and mix.

To make 0-, 10- and 20-ng/mL Fe solution, pipet 0, 20- and 20-mL of 0.1-µg/mL solution into 100-, 200-, and 100-mL volumetric flasks, respectively. Add 0.2 mL of ultrapure nitric acid for each 100 mL of solution to the 0 (Blank), 10- and 20- ng/mL standards. Dilute to volume with water. Higher standards can be made if required and if they are within a readable range.

Samples and ash blank must be diluted to the proper reading range. If a dilution of more than 10 times is needed, acidify with ultrapure nitric acid to contain 0.2% in the final volume. Carry out the same dilution with the ash blank as with the sample.

If an automatic sampling system is used, be sure to rinse the sampling cups with the blank, standard or sample solution to eliminate possible dust contamination.

Read each solution in duplicate in the following order.

Standard Blank	0
1st Standard	(10-ng/mL Fe)
2nd Standard	(20-ng/mL Fe)
Sample Ash Blank	
1st Sample	
2nd Sample	
etc.	

Read no more than 5 samples in duplicate before recalibrating; i.e., reread the standard blank, 1st standard, etc., after each 5 samples.

Follow the reading order as described in the iron analysis.

Vanadium Analysis

Set the instrument parameters and furnace conditions for vanadium.

The addition of aluminum to vanadium in the amount of 100 $\mu\text{g/mL}$ improves response and repeatability (see Note 3).

To prepare a 1000- $\mu\text{g/mL}$ Al solution, weigh 10.2 ± 0.01 g of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ into a one-liter volumetric flask.

To make 0-, 100- and 200-ng/mL vanadium standards, pipet 0-, 10- and 20-mL of 1-mg/L vanadium solution, respectively, into each of three 100- mL volumetric flasks. To each flask pipet 0.2 mL of ultrapure HNO_3 and 10 mL of the 1000- $\mu\text{g/mL}$ Al solution. Bring to volume with water and mix.

Check the response of the samples against that for the standards in the graphite furnace and make appropriate dilutions to bring them into reading range.

All final dilutions of the ash blank and samples should also contain 0.2 mL of concentrated HNO_3 and 10 mL of 1000 $\mu\text{g/mL}$ of aluminum per 100-mL final volume.

Follow the reading order as described in the iron analysis.

Nickel Analysis

Set the instrument parameters and furnace conditions for nickel.

To make 0-, 25-, and 50-ng/mL nickel standards, pipet 0-, 5- and 10-mL of 1-mg/L nickel solution, respectively, into each of three 200-mL volumetric flasks. Add 0.4 mL of concentrated nitric acid into each flask. Dilute to mark with water and mix.

Check the response of the samples against that for the standards in the graphite furnace and make appropriate dilutions to bring them into reading range.

All final dilutions of ash blank and samples over 10 times dilution, should contain 0.2 mL of concentrated HNO_3 per 100-mL volume.

Follow the reading order as described in the iron analysis.

Copper Analysis

Set the instrument parameters and furnace conditions for copper.

To make 0-, 10-, and 20-ng/mL copper standards, pipet 0-, 2- and 4-mL of 1-mg/L copper solution into each of three 200-mL volumetric flasks, respectively. Add 0.4 mL of concentrated pure nitric acid to each flask. Dilute to the mark with water and mix.

Check the response of the sample against that for the standards in the graphite furnace and make appropriate dilutions to bring them into reading range.

All final dilutions of ash blank and samples over 10 times dilution, should contain 0.2 mL of ultrapure HNO_3 per 100-mL volume.

Follow the reading order as described in the iron analysis.

Lead Analysis

Set the instrument parameters and furnace conditions for lead.

To make 0-, 25- and 50-ng/mL lead standards, pipet 0-, 5- and 10- mL of 1-mg/L lead solution into each of three 200-mL volumetric flasks, respectively. Add 0.4 mL of concentrated nitric acid into each flask. Dilute to the mark with water and mix.

Check the response of the samples against that for the standards in the graphite furnace and make appropriate dilutions to bring them into reading range.

All final dilutions of ash blank and samples over 10 times dilution should contain 0.2 mL of concentrated HNO_3 per 100-mL volume.

Follow the reading order as described in the iron analysis.

Sodium

Ion Exchange Column for Sodium Analysis

To remove all sodium from the organic solvents, prepare an ion exchange column using a 400-mm glass chromatographic column, a 500-mL separatory funnel and a ring stand of sufficient length, with clamps, for support. Clean the column and separatory funnel with hydrochloric acid followed by deionized water rinses and air dry. Fill the column with Amberlyst grade 15 resin leaving a 25-30 cm space at the top.

Alcohol Mixture for Sodium Analysis

Prepare a minimum of 500 mL of a 63% 1-butanol and 37% methanol mixture by volume. Ion exchange this mixture through the column previously described. Check the sodium content of the mixture in the flame of the atomic absorption instrument to make sure it is free of sodium. The first 50 to 100 mL of the exchanged alcohol mixture may be discarded until the alcohol is sodium free, i.e., there is no response for sodium when the alcohol is aspirated into the flame. If necessary, the alcohol mixture may be run through the column several times, but if the column is efficient, once should be enough. A new column may require conditioning with alcohol before it operates properly.

To the ion exchanged alcohol mixture add 3 mL of water per 100-mL volume. This is needed to pick up any of the inorganic sodium which may be present in the sample. Store in a Nalgene bottle that has been rinsed with sodium free alcohol.

Xylene for Sodium analysis

Normally the xylene is free of sodium, but it should be checked in the flame of the atomic absorption unit. If sodium is found, the xylene should be passed through the ion exchange column in the same manner as the alcohol mixture.

Sodium Analysis

The sodium analysis is a direct flame analysis requiring no ashing of the sample. Set the instrument to a wavelength of 589.0 nm with a slit setting 0.4 nm and use an air-acetylene flame with, if possible, a triple slot or Boling burner. Aspirate xylene as the rinse solution.

Heat the sample in an oven so that all waxy material is fluid and the sample flows freely. Shake the sample for at least 30 sec. By means of a Nalgene dropper and using a top loading balance, weigh into each of two Nalgene bottles (see Note 4) 4 ± 0.01 g of sample; less can be used if the sodium level is high. To the first bottle add 4 g of alcohol mixture and increase the mass to 20 ± 0.01 g with xylene. This is the sample dilution. To the second add 4 g of the alcohol mixture, 2 g of $0.5 \mu\text{g/g}$ Na in xylene and increase the mass to 20 g with xylene. This is the sample plus $0.05 \mu\text{g/g}$ Na dilution. Cap and shake the bottles until thoroughly mixed. Aspirate the sample into the flame of the AAS unit to check reading. If the reading is less than 0.10 absorbance units, no further dilutions are needed. If it is greater than 0.10 absorbance units, make appropriate dilutions until the reading is less than 0.10 absorbance units. The sample and sample plus standard should have the same sample weight of 4 g which is the largest amount of sample that can be used. In some cases, higher concentrations of sodium in the standards may be preferred.

CALCULATIONS

Calculate the concentration of nickel, vanadium, iron, lead or copper in the sample as follows:

$$\text{Element, ng/mL} = \frac{50 A - B_a/S - B_s CD}{W}$$

where:

A = average reading of sample, absorbance or chart units

B_a = average reading of sample ash blank, absorbance or chart units

B_s = average reading of standard blank, absorbance or chart units

C = concentration of standard, ng/mL

D = final dilution of reading sample = F/G

D = 1 where additional dilution is not required

F = final volume to which the aliquot is diluted, mL

G = aliquot from 50 mL volumetric flask, mL

S = average reading of standard closest to the sample reading, absorbance or chart units

W = mass of gas oil sample (usually equals 20), g

50 = volume to which the 20-g sample is diluted, mL

Calculate the concentration of sodium in the sample as follows:

$$\text{Sodium, } \mu\text{g/g} = \frac{20 (A/B - A) CD}{W}$$

where:

A = reading of sample, absorbance or chart units

B = reading of sample plus added standard, absorbance or chart units

C = concentration of sodium in the standard, $\mu\text{g/g}$

D = final dilution of reading sample = F/T

D = 1 for a sample which has not been diluted

F = final volume to which the aliquot is diluted, mL

T = aliquot from sample flask, mL

W = mass of gas oil sample (usually equals 4), g

20 = final mass of dilution, g

NOTES AND PRECAUTIONS

1. Sulfuric acid, electronic grade, is used instead of fuming sulfuric acid because all fuming sulfuric acid tested contained an appreciable amount of iron. A rust-free hood is needed to ensure accurate iron results.

2. Lead can be vaporized at temperatures above 600 C. Precautions should be taken to prevent the sample from heating above this temperature.

3. Vanadium atoms ionize in the flame as well as in the furnace. Aluminum added to solutions of vanadium reduces ionization, and 100 $\mu\text{g/mL}$ was chosen as an optimum concentration. A higher aluminum concentration might work a little better, however solids buildup in the furnace becomes a detriment, thereby shortening graphite tube life.

4. Sodium is virtually everywhere so precautions should be taken to reduce contamination. All Nalgene plasticware should be rinsed with methanol that has been ion-exchanged. Minimum dilutions and handling are recommended to reduce erroneous sodium results.

PRECISION

Based on 7 replicate determinations shown, the estimated standard deviations (esd) were calculated for the elements listed in the table below. Duplicate results by the same operator should not differ by more than the allowable difference shown (95% probability).

Element	Number of Replicates	Concentration, $\mu\text{g/mL}$	esd, $\mu\text{g/mL}$	Allowable Difference, $\mu\text{g/mL}$
Fe	7	1.81	0.027	0.100
Ni	7	0.18	0.013	0.045
V	7	0.77	0.027	0.097
Cu	7	0.16	0.012	0.042
Pb	7	0.17	0.023	0.081
Na	6	0.24	0.029	0.105

TIME FOR ANALYSIS

The elapsed time for ashing and sample dilution of a single sample is 24 hours. An additional hour is required for each metal determined. Therefore, the total elapsed time to determine all 6 metals in a single sample is about 30 hours. The labor requirement for all 6 metals in a single sample is about 7 hours.

SUGGESTED SUPPLIERS

Angstrom, Inc., P.O. Box 248, Belleville, MI 48111
 Corning Glass Works, Corning, NY 14830
 Fisher Scientific Co., 1600 W. Glenlake Ave., Itasca, IL 60143
 J. T. Baker Chemical Co., 222 Red School Lane, Phillipsburg, NJ 08865
 Perkin-Elmer Corp., Instrument Div., Main Ave., Norwalk, CT 06856
 Rohm and Haas, Independence Mall West, Philadelphia, PA 19105
 Sargent-Welch Scientific Co., 7300 N. Linder Ave., Skokie, IL 60077
 SPEX Industries, Inc., P.O. Box 798, Metuchen, NJ 08840