OPERATING INSTRUCTION

VREELAND SPECTROSCOPE MODEL 7

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SPECTREX CORPORATION

Standard Spectrograms in Alphabetical Order

			Film No.
Aluminum	(A1)		4
Aluminum Bands			75
Barium	(Ba)		46
* Beryllium	(Be)		50
Bismuth	(Bi)		8
Carbon Bands	(C)		74
Cadmium	(Cd)		14
Calcium	(Ca)		48
Calcium Bands	(Ca)		76
Calcium Fluoride	(CaF)		77
Caesium	(Cs)		41
Cerium	(Ce)		67
Chromium	(Cr)		21
Cobalt	(Co)	i	23
Cobalt	(Co)	e	53
Columbium	(Cb)	i	26
Columbium	(Cb)	e	59
Copper	(Cu)		12
Europium	(Eu)		72
Fluorine Bands	(Fl)		77
Gadolinium	(Gd)		71
Gallium	(Ga)		3
Germanium	(Ge)		5
Gold	(Au)		11
Hafnium	(Hf)		32
Indium	(In)		2
Iridium	(Ir)		35
Iron	(Fe)	i	22
Iron	(Fe)	e	52
Lanthanum	(La)		66
* Lead	(Pb)		7
Lithium	(Li)		45
Magnesium	(Mg)	i	18
Magnesium	(Mg)	e	49
Manganese	(Mn)	i	16
Manganese	(Mn)	e	55
* Mercury	(Hg)		13
Molybdenum	(Mo)	i	19
Molybdenum	(Mo)	e	56
Neodymium	(Nd)		69
Nickel	(Ni)	i	25
Nickel	(Ni)	e	54

			Film No.
* Osmium	(Os)		36
Palladium	(Pd)		37
Potassium	(K)		43
Platinum	(Pt)		34
Praseodymium	(Pr)		68
Rhenium	(Re)		40
Rhodium	(Rh)		38
Rubidium	(Rb)		42
Ruthenium	(Ru)		39
Samarium	(Sm)		70
Scandium	(Sc)		64
Scandium Bands	(Sc)		80
Silver	(Ag)		9
Sodium	(Na)		44
Strontium	(Sr)		47
Strontium Bands	(Sr)		78
Tantalum	(Ta)		27
Tantalum	(Ta)	i	60
Thallium	(Tl)	e	1
Thorium	(Th)		30
Titanium	(Ti)	i	17
Titanium	(Ti)	e	61
Tin	(Sn)		6
Tungsten	(W)	i	28
Tungsten	(W)	e	57
Uranium	(U)		29
Vanadium	(V)	i	25
Vanadium	(V)	e	58
Yttrium	(Y)		65
Yttrium Bands	(Y)		79
Zinc	(Zn)		15
Zirconium	(Zr)	i	31
Ziron	(Zr)	e	62

Angstrom Scales	10, 33, 60E
Key Lines	20, 60D
Key Lines (singlet)	51
Key Lines (doublets)	
Key Lines (triplets)	63
Key Lines (4 to 7 line groups)	73

i = Identification film
e = Elimination film
* = Danger Toxic. Use fume hood

Melting Points of Elements on Films

	Melting Point °C	Boiling Point °C
Aluminum	659.7	2057
Barium	725	1140
Beryllium	1278	2970
Bismuth	271.3	1560
Carbon	>3550	4200
Cadmium	321	767
Calcium	842	1240
Cesium	28.5	670
Cerium	804	1400
Chromium	1890	2480
Cobalt	1495	2900
Columbium	2500	3700
Copper	1083	2336
Europium	1150	---
Gadolinium	---	---
Gallium	29.78	1983
Germanium	958.5	2700
Gold	1063	2600
Hafnium	1700	>3200
Indium	156.4	2000
Iridium	2454	>4800
Iron	1535	3000
Lanthanum	826	1620
Lead	327.43	1336
Lithium	186	1107
Magnesium	651	1900
Manganese	1260	356.58
Mercury	-38.37	4800
Molybdenum	2620	2900
Neodynium	840	>5300
Nickel	1455	2200
Osmium	2700	760
Palladium	1549.4	4300
Potassium	62.3	2500
Platinum	1773.5	700
Praseodymium	940	2700
Rhenium	3167	
Rodium	1966	2400
Rubidium	38.5	1950
Ruthenium	2450	880
Samarium	1300	1150
Scandium	1200	4100
Silver	960.8	1457
Sodium	97.5	4500
Strontium	774	
Tantalum	2996	
Thallium	302	
Thorium	1845	

Melting Points of Elements on Films

	Melting Point (°C)	Boiling Point (°C)
Titanium	1800	3000
Tin	231.89	2270
Tungsten	3370	5900
Uranium	1130	---
Vanadium	1710	3000
Yttrium	1490	2500
Zinc	419.47	907
Zirconium	1857	2900

Elements without Master Films Which Have Visible Lines
(Use Angstrom Scale For Identification)

Element	Wave Length	
Dysprosium	4000.4Å	4211.7 Å
Erbium	5414.6 Å	
Holmium	4882.9 Å.	
Lutecium	5476.6 Å	
Ruthenium	4554.51 Å	6923.2 Å
Terbium	4276.71 Å	4278.5 Å
Thulium	4242.1 Å	4359.9 Å
Ytterbium	6667.8 Å	6799.6 Å

Elements Identified With UV/IR Modification

Elements	Wave Length	
Antimony	2528.5 Å	
Arsenic	2860.5 Å	
Boron	2496.7 Å	2497.7 Å
Silicon	2881.5 Å	
Tellurium	2383.2 Å	2385.7 Å

Minimum Detection Limits of Elements identified on the Vreeland Spectroscope

These figures have to be used with some reservation due to the variability of matrix effects.

	%		%
Aluminum	.003	Molybdenum	.001
Barium	.003	Neodymium	.1
Beryllium	.2	Nickel	.01
Bismuth	.01	Osmium	.1
Carbon	No Standards	Palladium	.01 -.05
Cadmium	.05	Platinum	.1
Calcium	.005	Potassium	.05
Calcium Fluoride	.5	Praseodymium	.5
Cesium	.5	Rhenium	.1
Cerium	.05	Rhodium	.1
Chromium	.002	Rubidium	.1
Cobalt	.02	Ruthenium	.1
Columbium	.2	Samarium	.1
Copper	.001	Scandium	.01
Europium	.5	Silver	.003
Gadolinium	.05	Sodium	.0005
Gallium	.01	Strontium	.005
Germanium	.05	Tantalum	.1
Gold	.05	Thallium	.001 -.01
Hafnium	No Standards	Thorium	.5
Indium	.01	Tin	.005
Iridium	.1	Titanium	.002
Iron	.0015	Tungsten	.05
Lanthanum	.05	Uranium	.1
Lead	.0015	Vanadium	.002
Lithium	.002	Ytrium	.01
Magnesium	.003	Zinc	.003
Manganese	.005	Zirconium	.01
Mercury	.1		

Note: 1 part per million = 0.0001%
100 parts per million = 0.01%

Section General I

General Description 1.

A system of spectroscopic analysis is described which eliminates measurement of wave lengths and reference to tables by direct comparison of the spectrum of the sample with master spectra of the several elements. Master films are built into a spectroscope having an optical system that projects two continuous spectra adjacent to the spectrum of the sample and the master films are illuminated in their true colors for comparison, line by line, with the observed spectrum. Elimination films select out the lines of major constituents so the lines of other constituents are apparent for comparison with identification films. The master spectra are classified by index charts.

The Spectroscope operates on 115 volts alternating current. The sample is placed on a disposable refractory hearth adjustably mounted below a horizontal arc. Progressive heating permits successive excitation of the spectra of the component elements and facilitates their identification.

Optical Specifications 2.

Concave diffraction grating. Focal length 24 cm. Instrument dispersion 40.3 angstrom/mm. Capable of analyzing all elements with lines in the visual range 4000 to 7000 Å.

Applications 3.

a. **Mineralogy:** The Vreeland Comparator Spectroscope makes possible the rapid and precise identification of all the common and most of the rare elements occurring in minerals. The instrument has a unique place in the field of mineralogical investigation and is being used by national geological laboratories, state bureaus, assay and mining companies and by individual hobbyists.

Field Work: Coupled to a suitable motor-generator it is ideal for field surveys where quick, precise checks have to be made and where expensive, time-wasting analysis at headquarters is to be avoided.

b. **Metallurgy:** The Spectroscope can be used to give rapid and accurate identification of all the common metals and their components. Such applications as the identification of stainless steels and the impurities in metallurgical processes illustrate its wide adaptability.

c. **Inorganic Chemical Analysis:** All the major inorganic elements have master films and very rapid qualitative analysis can be obtained of solid or liquid samples, avoiding wet analysis.

d. **Teaching:** *High School* Level – The Spectroscope is ideal to demonstrate the optical principles of diffraction and the origin and interpretation of spectra due to the simplifying effect of seeing the direct comparison feature.

College Level – Its use is as above but also as a tool of the applied science of examining materials.

Post-Graduate Level – It is ideal as a low-cost tool for detailed analysis in advanced research.

Departments most likely to use the Spectroscope:

1) **Geology** – for rapid mineral identification where students can do their own analysis.
2) **Chemistry** – for freshmen chemistry; to either augment or replace wet analysis and as an introduction to spectroscopy.
3) **Biology** – to identify trace inorganic elements in leaf compressions, soils, etc.
4) **Physics** – to demonstrate the origin and interpretation of spectra. The solar scope attachment provides solar spectra.

e. **Crime Detection:** It provides quick identification of the trace elements in criminal samples.

f. **Air and Water Pollution:** Air samples can be collected in a high volume filter and ashed. Water samples are evaporated to a residue. This greatly lowers the sensitivity limits of analysis.

Power Requirements 4.

The instrument is designed to operate from a 115V AC 50/60 cycles per second source (1000 watts). If only 220V AC power is available a special modification can be provided on request. Always use a 3-prong socket.

CAUTION: The instrument is not designed to operate from a DC Source.

Accessories 5.

300 Refractory Hearths of special design. More of these can be purchased from Spectrex Corporation.

10 Sample Graphite Squares to fit onto hearths to improve sample burning and facilitate reuse of hearth. (See Page 10). More can be purchased from Spectrex Corporation.

50 Carbon Electrodes of special design and purity. More of these can be purchased from Spectrex Corporation.

1 Copy Reference Chart(s) for Master Films. Charts 1 and 2 have been specially mounted to withstand rough wear and provide rapid location of any sections of the Master Films. The charts are located on either side of a single sheet. Chart 1 lists the elements in film order as found on the left hand and right hand Master Films. Chart 2 lists these same elements alphabetically.

1 Set Special Tweezers for handling hot ceramic hearths.

1 Camera Adaptor Tube (Item 42 exploded view, Page 33). The instrument may arrive with the black-anodized adaptor tube screwed onto the eyepiece. It should be removed for normal, visual operation.

1 Copy Operating Instruction Manual.

1 Dust Cover.

1 Protection cap (red, rubber) to be used on front electrode if instrument is connected with a 2-prong plug.

1 Wire Brush for cleaning out electrode holes in arc block to prevent carbon build-up. Clean every six (6) months.

1 Set (4) Allen Wrenches.

Toxicity 6.

It is strongly recommended that a fume hood or exhaust vent be used with the instrument. A simple flexible 6″ diameter tube, suspended 4″ over the arc block chimney and led out to a fan exit near the ceiling is adequate. The following elements are toxic and should not be vaporized without a fume hood: Lead, Mercury, Osmium, Nickel and Beryllium.

Procedure for Replacing Objective Lens 01 7.

(Cat. 01900 #10 Exploded View).

After two or three years or when specimens which splatter are constantly used, the objective lens, which collimated the light from the arc, will become pitted and should be replaced. This is easily done, following the procedure below:

a. Remove front cover by unscrewing 10-24 flathead screw on right hand surface of cover and two vertical screws accessible from underneath the instrument through 2 clearance holes. (Use Allen Wrenches provided). Also remove arc block control knobs.

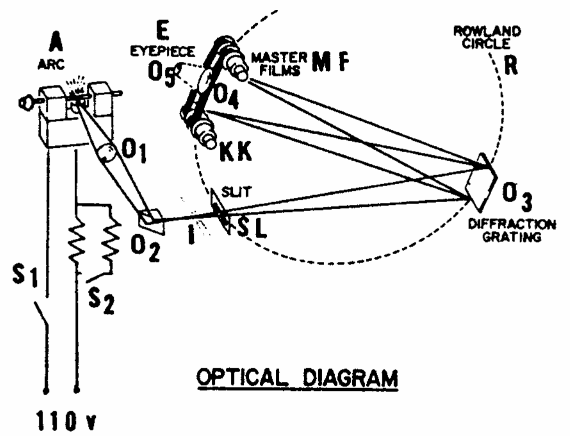
b. Without loosening the periscope tube clamps (#12 exploded view), unscrew locking sleeve (#11) and remove pitted lens (#10).

c. Insert new lens (available from Spectrex) and tighten locking sleeve.

d. Refit front cover and tighten screws.

Section Operation II

The Apparatus (see Diagram) 1.



A is an arc holder, with a disposable refractory hearth on which the sample is placed. S1 is a switch for turning the arc on and off. E is an eyepiece by which the spectrum is magnified and viewed. MF are mylar films on which the master spectra are recorded, placed on both sides of the observed spectrum for direct comparison. KK are knobs for turning the spools on which the master spectrum films are wound.

The optical system is simple. The parts are rigidly mounted on pedestals on a substantial base casting and do not require adjustment. The spectrum is formed by a concave reflection grating of short focus, which not only makes the apparatus compact and simple, but forms a spectrum of great brilliance which is sensitive to elements present in small amounts. The focal length is chosen to give resolution sufficient for the separation of any elements likely to be found in mineralogical work, including minor constituents in irons which require care and precision when the ordinary spectroscopic methods are used. *

Referring to the drawing above, it will be noted that the points of the carbon electrodes of the arc are arranged in a horizontal plane, not vertically as in the usual spectroscopes and spectrographs. The sample is not placed in a hollow of the electrode, as is customary, but on a dispensable refractory hearth below the electrodes. The hearth adjacent to the electrodes has the effect of conserving and concentrating the heat of the arc. The hearth has a vertical adjustment for height, which permits control of the rate of "burning" of the sample, so that the spectra of the several component elements may be excited progressively and studied one by one instead of mingled together in confusion.

* See Appendix V. 2.

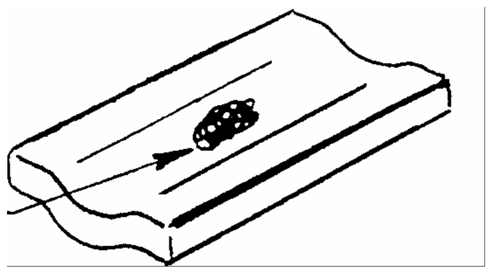
The alternating current in the arc is stabilized by fixed ballast which does not require adjustment. For exceptional samples of highly refractory material an additional ballast is provided and snapped on by a switch, S2, giving higher temperature in the arc. The optical system is designed to bring the spectrum of the sample to a focus in a curved band 3 mm. wide in the slot between the films MF, across which the characteristic lines of the component elements appear, like rungs of a ladder, magnified by the eyepiece E. The lines are seen in the full brilliance of their colors against a dark background, free from dimming by any continuous spectrum.

The optical system also projects at the same time two continuous spectra, on opposite sides of the line spectrum of the sample and immediately adjacent to it. The films, MF, carrying the master standard spectra and arranged parallel to and on opposite sides of the spectrum of the sample, are illuminated by the two continuous spectra. The master standard films are photographic positives so that the lines of the standards, each illuminated by the correct part of the spectra, appear in their true spectral colors. They may thus be compared directly with the corresponding lines of the observed spectrum. When the spectrum of the sample J and the standard are matched, the lines appear continuous and unbroken across the spectrum of the sample and the master film, so that any departure from the standard Is immediately apparent.

By this means, not one, but all the lines characteristic of an element can be matched against the standard with a precision greater than could be expected by the measurement of the wave lengths of the lines, one by one, by any feasible scale. By the multiple coincidences of all the lines of the spectrum of an element, its identification is assured with precision and positive ness. If there are any lines in the spectrum that do not match, they are recognized at once as belonging to another element or elements, which are thus marked for identification in their turn.

The matching of the standard to the spectrum is assured by using the two 0 lines of sodium as a datum, to which the film is adjusted by means of the knobs KK. The 0 lines are recorded on each master standard film immediately under the emblem for the element. The 0 lines are exceedingly sensitive and are produced by less than 0.001% of sodium in the electrodes. See later for detailed use of Master Films.

Preparation of Samples 2.

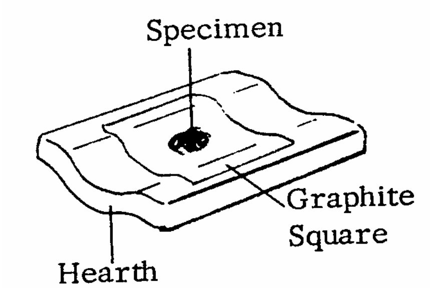


Pulverize about 25 milligrams of the sample in a mortar and place in a small pile in the center of a ceramic hearth. Materials and ores rich in chromium, iron, copper and similar metals will often melt down into masses too conductive and too large for vaporization, or will bubble and boil and blur the spectrum. It may be necessary to dilute the pulverized sample with quartz or carbon powder. Metal can often best be analyzed in the form of filings.

Loading of Samples 3.

a. **Normal Way.** The ceramic hearth is disposable. A fresh one should be used for each sample. It is loaded and then placed on the small aligning platform between the electrodes, with groove running parallel to the electrodes. It has been specifically designed for the Vreeland spectroscope and is reasonable pure. However, after continuous burning, Titanium, Calcium, Magnesium and Sodium lines will appear and are trace contaminated.

To eliminate these traces and provide a steadier Burn, graphite squares are available from Spectrex which fit onto the hearth as per sketch and Facilitate reuse of ceramic hearth. A sample Packet of 10 squares are provided with your Spectroscope.



b. **Alternative Method** (using special carbon cups available from Spectrex Cat. #01310). Load the powdered specimen into 1/8″ diameter cup and place into 1/8″ diameter hole drilled in standard electrode. This provides exhaustive specimen burning and is particularly recommended for Quantrex work. Alternatively, mount carbon cup vertically in 1/8″ diameter hole drilled in fixed hearth and feed it up into arc as with ceramic hearth. A special "carbon cup kit" consisting of 100 carbon cups, loading platform, ramrod, loading funnel, fixed hearth drilled with 1/8″ diameter hole and 3 electrodes drilled with 1/8″ diameter hole can be ordered from Spectrex.

c. **Electrode Contamination**
The electrodes are cored, micro-projector carbons and contain sufficient sodium trace to provide a steady sodium doublet. This gives a reference point for the alignment of each film. Pure, high-grade carbon or graphite carbons would not give a steady arc on A.C. current.

Because of the separate specimen feed, it does not matter that the electrodes are not ultra pure. The eye can easily discriminate between the lines from the electrodes and the lines from a sample as these latter will only appear when the sample is raised up into the arc.

Step-by-Step Operating Instructions 4.

a. Clean electrodes if they have been used for previous analysis. Sample traces are inclined to get deposited on the electrode tips. Simply break off the tips with a pair of long-nosed pliers. (It is not necessary to sharpen the electrodes. They can be fed into arc block in the blunt state and are self sharpening as the arc burns.) For new electrodes, wait until the iron on the tips has burnt off before burning specimen. Use wire brush to clean electrode holes, if necessary.

b. Put awning (Item #46, exploded view) to "down" position and place prepared sample in a refractory hearth and locate beneath electrodes.

c. By turning electrode feed knob counter clockwise unscrew electrode block posts until they are at their maximum distance apart.

d. Flip up electrode alignment lever (Item #66, exploded view) and load electrodes so that tips touch each side of the lever. The rear electrode is driven into the arc block post through the safety sleeve by engaging the electrode retractor. (Item #63).

e. Place red rubber sleeve provided over forward, protruding electrode for extra safety. (See safety features, Page 30).

f. Turn electrode feed knob half turn clockwise and then remove electrode alignment lever from between electrode tips.

g. Position black awning in upright position to prevent light leakage.

h. Plug power cord into 3-prong receptacle and switch on instrument with switch S1.

i. Screw the electrodes together by turning the electrode feed knob X clockwise until the arc is struck. Set the arc gap at 1/8".

j. Look through the eyepiece to ensure correct gap and alignment. The continuous spectra from the arc tips should illuminate the films while the area between should be darker. The lines seen there, at this stage, represent carbon bands from the electrodes and sodium doublet.

k. Refer to either the alphabetical or numerical Element Index Chart and note the spectrograms of the elements you suspect are in the sample.

i. If you have no idea of what the sample contains, note the spectrograms on the key lines film – number 20 on right hand film or 60D on left hand film

l. Observing films, turn to film 20 or the required spectrogram. It is not necessary to look through the eyepiece to see the film numbers. They are clearly visible as the film slides over the polished steel film holder plate.

m. Align the sodium D lines, a couplet just beneath the emblem for the element, indicated by a heavy arrow, with the D lines seen between the films. Sodium always shows as it is in the electrodes. Now the film spectrogram is aligned.

n. Slowly turn the “specimen feed” knob clockwise, raising the sample just below the arc. Watch the “burn” through the dark “welders” glass.

o. Note the lines which appear and see if they line up with the key lines on film 20 or other spectrograms in position. Scan the entire visible spectrum by sliding the eyepiece up and down.

p. Lower the specimen. If the specimen feed mechanism is too loose and falls of its own weight, tighten it by tightening the 8-32 button head screw in the center of the arc block side plate (Item #65, exploded view).

q. Repeat process with other spectrograms until all elements indicating lines in the visual range have been identified. Note: The more volatile elements will be the first to emit spectral lines.

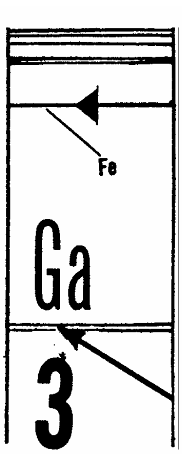
r. To obtain a higher arc temperature, use switch S2 to put a second resistor in parallel with the first and so permitting more arc current. The temperature goes from 2000°C to about 3000°C. Do as much analysis using “low heat” as possible.

s. Maintain arc distance at 1/8″, periodically screw electrodes towards each other as they burn. This maintains good film illumination.

t. If arc is not central, adjust electrode nearest you by hand in conjunction with the use of the electrode feed control knob. The rubber sleeve provided should be placed over the forward electrode as a precaution.

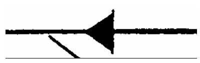
u. To remove hot sample, use tweezers provided and place onto unpainted platform behind electrode block.

Use of Films 5.



The following is an example of a typical spectrogram mounted on the mylar films:

Note that the sodium doublet which is used to align the spectrogram is always immediately under the element emblem and is marked with a heavy arrow.

This symbol  represents the line(s) which has maximum sensitivity.

The diagonal lines, with element symbols attached, indicate lines of these particular elements which lie very close to the line on the spectrogram and hence could be mistaken for it. They speed up line identification since, if you are not quite sure if a line matches one on the spectrogram, an immediate indication is given of other spectrograms to turn to as giving possible match.

Elements, such as iron, which have many lines appearing in the visual spectrum, can obscure lines of other elements and cause confusion. These elements are each provided with an "elimination film" (signified by a small "e" immediately above the element symbol and D lines) which contains **all** its visible lines. This can be lined up on the left of the sample lines and so used to "eliminate" all the lines of the sample belonging to the complex spectrum. The lines which do not align must, therefore, clearly belong to other elements and the right hand roll can be used to identify them.

Angstrom Scale Films (Spectrograms 10, 33 and 60D) 6.

These are useful references when the wave length of any particular line or group of lines is required. It provides an additional check in identifying elements and can be used in conjunction with standard wave length tables such as the M.I.T. Tables, those in "Chemical Spectroscopy" by W.R. Brode, (John Wiley & Sons) and in "Manual of Spectroscopy" by T.A. Cutting (available from Spectrex).

Semi-Quantitative Analysis 7.

The use of the "elimination" and "identification" spectrograms. The instrument does not pretend to be a quantitative instrument. However, it is adapted for the semi-quantitative estimation of the relative amounts of the elements present in a sample by comparison of the spectrum with the identification and elimination films. As stated in Section II. 5 the elimination film includes all the visible lines of an element. The identification film shows only the major lines. It is indicated of the film by a small "I" immediately above and to the right of the element symbol and sodium D line.

In general, it may be said that a major constituent should show all the lines of the elimination film; a minor constituent, the strongest of those lines; a small percentage, the lines of the identification film; and a trace, only the most prominent lines. One may thus distinguish between essential components of a mineral, minor constituents or isomorphous substitutions and impurities.

Moreover, with the use of the arrowed lines (of maximum sensitivity), rough semi-quantitative analysis is obtained. Finally, with the Quantrex attachment, specific line intensities can be obtained. (See Section III. 2.).

Key Lines (Films 20, 60D, 51, 63 and 73) 8.

Films 20 and 60D have the key line or lines for all the elements analyzable by the Vreeland Spectroscope. They are useful films to turn to if you have no idea as to what is in the sample.

Film 51 has the major, **singlet,** key lines for rapid identification of unknowns.

Film 63 has the major **doublet and triplet** key lines for rapid identification.

Film 73 ha the major **quadruplet, quintuplet and septuplet** key lines.

The Camera Adaptor 1.

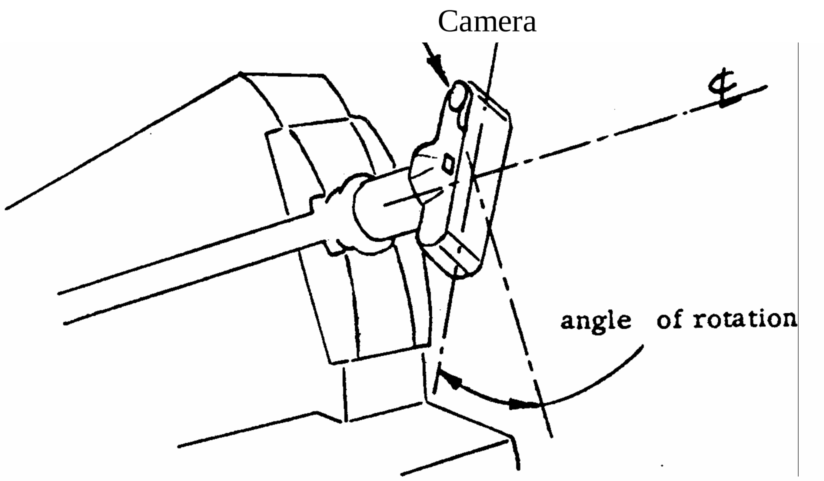
The adaptor tube (Item #42, exploded view) is a rapidly fitted, knurled, threaded tube mating any interchangeable lens camera to the spectroscope.

One end has a standard thread connecting it to the eyepiece tube while the other has a standard 49 mm. thread. An adaptor ring can be purchased from any camera store which connects your camera to this threaded end.

a. Using 35 mm. Camera

1) Vertical alignment of camera
When the camera is fully screwed onto eyepiece, it may not be in a suitable, vertical position....

This adjustment, however, **should not** be made if your instrument is already set for a Quantrex attachment.



a) Note the angle of rotation needed to make camera vertical.
b) Unscrew the three set screws (at 120° to each other) holding the eyepiece tube to the eyepiece casting.
c) Rotate the tube, adaptor and camera until the camera is vertical.
d) Tighten the three set screws.

Further Points to note 2.

A single lens reflex camera is recommended as the spectrum to be recorded can be examined at the moment of shooting.

A wide angle lens provides maximum field of view of the spectrum

Focus the camera while viewing the spectrum. In the case of the Pentax you will find the setting to be almost at infinity. The eyepiece optics act as a close-up lens.

In the field of view is restricted to a small area of the spectrum, even with a wide angle lens, unscrew the spectroscope eye lens a distance of ¼″ to ½″.

Use a standard cable release mechanism.

Types of film and exposure. Most quality color and black and white films give good results. Excellent results have been obtained with Kodachrome X with an exposure time of 3 seconds in the yellow-green range. This film is ideal for color slides while Kodacolor-X would best be used for color prints. Kodachrome II is a slower film. A good middle range slide can be obtained with a ten second exposure. We recommend 12-15 seconds in the blue and eleven to twelve in the red, with further modifications depending on the specific analysis being run.

For high precision work, black and white film is recommended. Any medium-speed, fine-grain film is adequate though Panatomic X or Kodak Direct Positive films are preferred. Exposure time with both types is 5-10 seconds when the camera is set for spectra with a center at about 5500 angstroms. Development is in D-10 for 6 minutes at 68°F. This can then be blown up to a dispersion of 5 angstroms per mm. with no difficulty and provides the kind of resolution the multi-thousand dollar spectrographs give.

Applications 3a.

Teaching. Most chemistry and geology departments of colleges and universities already have a 35mm. camera. The color slides resulting from Kodachrome film give a very exact duplication of the colors and spectra as seen through the eye-piece and can be projected for a whole class to see. Thus, students can be shown exactly how the Vreeland Spectroscope analyzes specimens without the need for an individual demonstration for each student.

Analysis with Slides. A library of slides of standard specimens with varying percentages of the elements can be collected. Slides of unknowns can then be projected side by side with these standards and line intensities compared with a photometer. One user working in semi-quantitative analysis writes:

"Generally we do not find it inconvenient to use a whole roll of 35 mm. film before analyzing the pictures. If enough sample is available, we run a visual first and then make the photographs for detailed examination. We find that in a complex sample containing both low volatile and refractory metals, it is necessary to make a series of exposures to cover the complete range of times of volatilization. Thus, one analysis will consist of several photographs of each of three spectral ranges. We find that three overlapping photographs will cover fairly well the range covered by the Vreeland Spectroscope."

Semi-quantitative work. The success of this depends to a great extent on the skill and experience of the operator. An assumption of standard processing procedures must be made when comparing line intensities. For best results the processing for standards and unknowns should be done by the same laboratory at the same time.

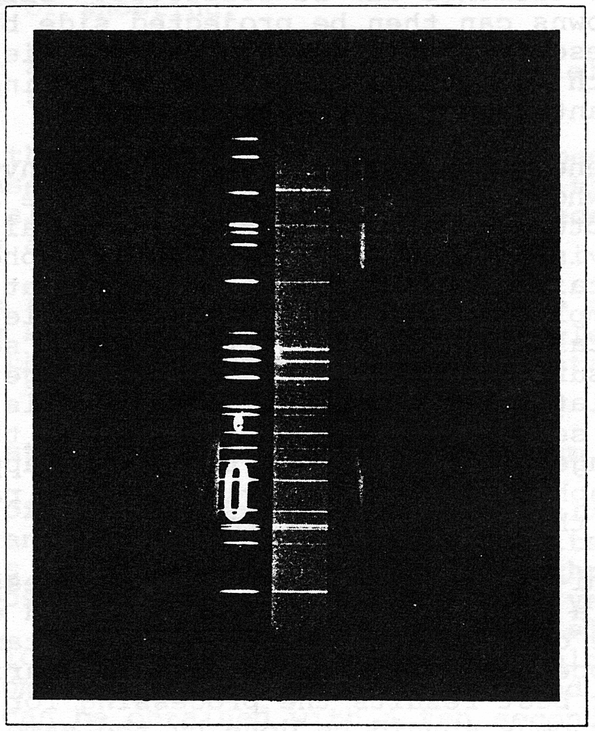
Using Standard SX-70 Polaroid Camera 3b.

1) Procedure

Attach the camera to the spectroscope using the adaptor bracket available from Spectrex (Cat. #66320). The advantage of this approach is that it provides a Polaroid single-lens-reflex system.

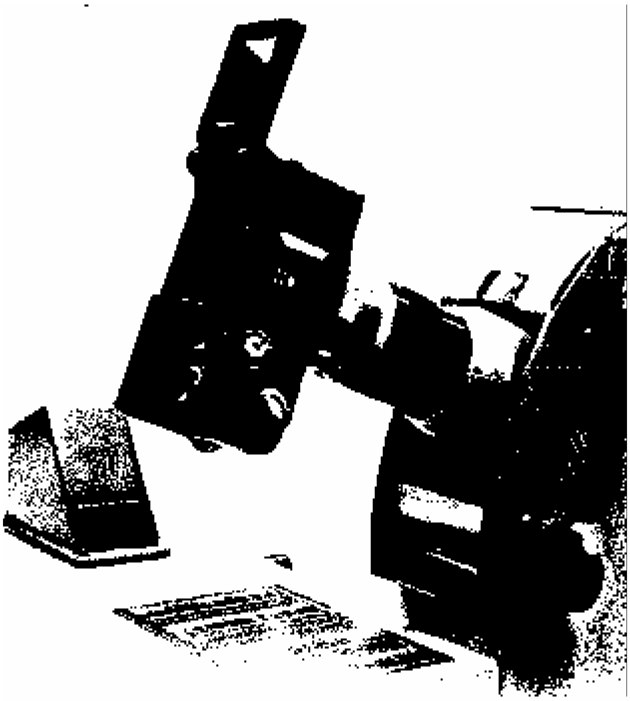
2) Major Application – Teaching

Immediate, permanent records. Students can take their own Polaroid pictures and insert them directly into their laboratory books. A number of chemistry departments in colleges are using the Vreeland Spectroscope to augment their wet analysis course. Students can confirm their own wet analysis course. The wet analysis findings can be confirmed by simple spot checks with the Vreeland Spectroscope. The instrument does not replace wet analysis but demonstrates this alternative method for solving analysis problems. It is an ideal introduction to practical spectroscopy. The student's Polaroid pictures are the permanent proof of his findings. It is difficult to dispute such inescapable evidence!



Ever since the personal computer came into being there have been constant requests from our Vreeland spectroscope users to bring the spectral lines from the Vreeland into their computers for more deliberate and thoughtful analysis. In essence they wanted their spectroscope to become a broadband spectrograph able to input images and data into their personal computers.

So Spectrex is delighted to announce that we have now found a way to satisfy that need by use of a high quality digital camera and a special edition of Photoshop 5; while retaining all the advantages of the original machine. The digital camera we have selected, the Canon G1 with 3.34 million pixels, has a threaded body that allows precise, solid positioning so that the background spectral lines created by a sample arc can easily be compared to a background image in Photoshop. A custom machine fitting is supplied with this upgrade to connect the Canon G1 to the Vreeland eyepiece.



The spectral lines created by the grating are accurate to about 1 Angstrom and in the Vreeland Model 7 can be ready by eye to about 5 Angstroms. In normal operation, the sample is lifted into the arc and goes through a rather rapid distillation so spectral lines due to the sample are of short duration time wise. While this is satisfactory for the identification of an individual element, it is difficult to search the entire visible spectrum and get consistent readings for the five frames that are visible at any one time. Accordingly, provision has been made in this computerized upgrade of the Vreeland Model 7 to achieve a much longer sample burn thus making sample spectral lines visible for minutes rather than seconds. This is realized by inserting the sample in the outboard carbon, which has been drilled with a custom jig. Of course the elevator and porcelain boat remains available should that method of burning a sample be desired.

Once the spectrum lines are imported into Photoshop 5 LE they can be studied at leisure and read down to one pixel or 1/3 Angstrom under ideal conditions. This level of accuracy requires the use of a special Angstrom scale, which is supplied as a part of the upgrade, and the zoom capability of Photoshop. The net effect is that this upgrade allows careful study of sample spectral lines with a potential increase in accuracy of over 10 times. In terms of eliminating background lines this is a great improvement.

To achieve the level of accuracy noted above very large image files are created and the user's computer must have sufficient memory in either the hard drive or, preferably, a read-write CD for the storage of retained images. For example to store the entire visible spectrum in maximum detail requires approximately 10 megabytes.

Other System requirements are:

1. Windows 95, 98, NT4.0 (Service Pack 3 or later required)
2. Pentium 133 Mhz or better
3. Ram 64 MB or better
4. USB (Limited to preinstalled Windows 98 or Windows 2000
5. RS-232C serial or com port (If USB connection is not available)
6. Free hard disk space-375 mb plus whatever image storage is desired
7. Recommended display – 1078x600 pixels, high color 16 bit or better
8. Read-Write CD is not a requirement but very helpful

i) The Ultra Violet/Infra Red Modification

The changes to your Vreeland Spectroscope to extend the spectral range from 4000Å - 7000Å to 2500Å - 8500Å can be made by you and only takes a few minutes. Spectrex would send you four items:

a. Quartz collimating lens.

b. Special film holder.

c. Special quartz optics for eyepiece arm.

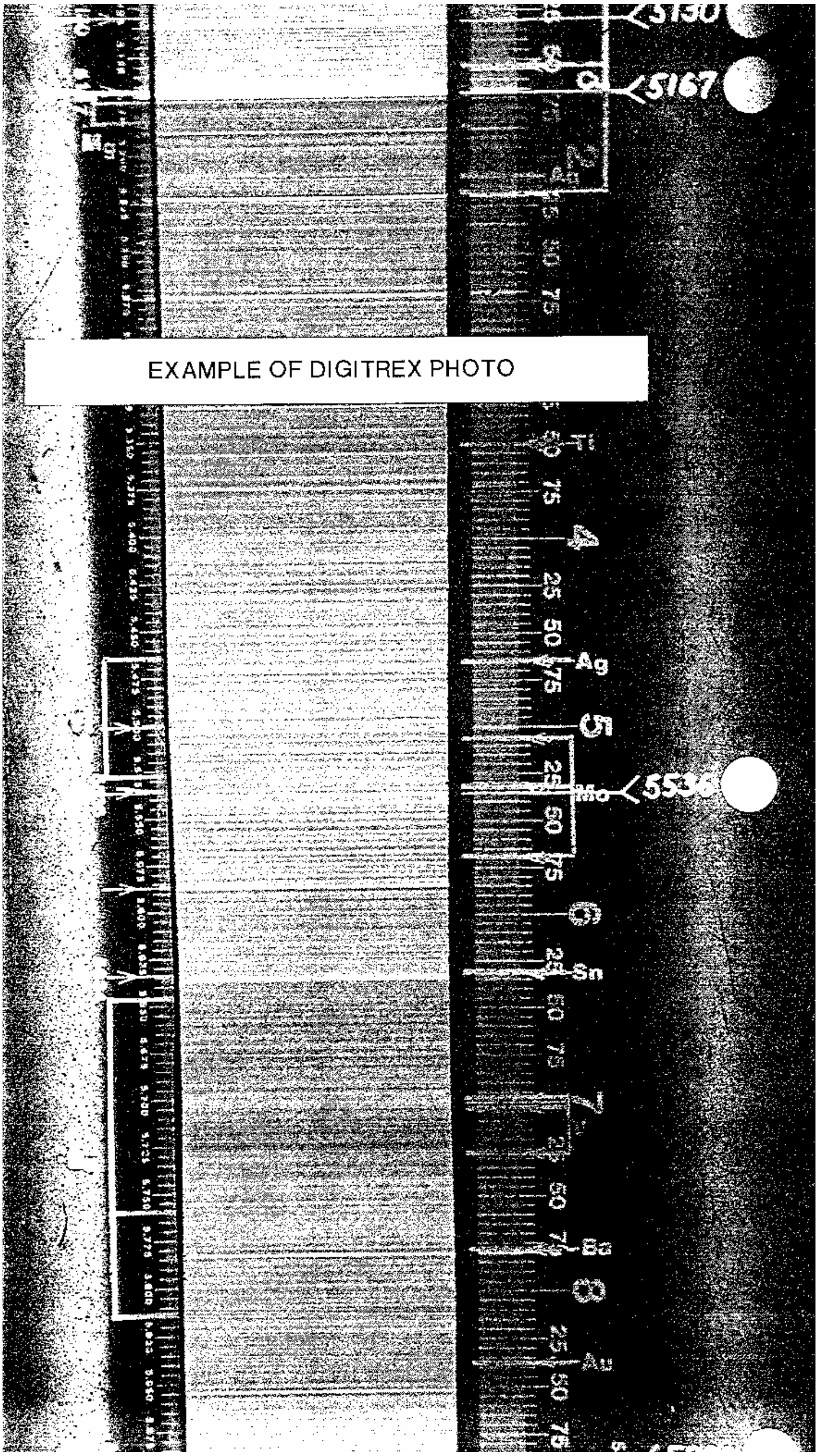
d. 35mm camera back and adaptor tube.

ii) The Solar scope Attachment

This is a system of mirrors and lenses which are so arranged to channel the sun's rays to come to focus at the slit of the spectroscope. Viewing the resulting spectra by eye or by camera clearly shows the absorption spectrum of the sum. The wave length of each line can be measured using the angstrom scale on the right hand master film.

iii) Video Spectra Scan Closed Circuit TV Attachment

An easily attached camera and monitor permit the simultaneous study of a spectrum by a whole class of students or a team of analysts.



EXAMPLE OF DIGITREX PHOTO

Section Principles of Operation IV

Spectroscopic Analysis 1.

Newton showed that white light is composed of light of all colors of the visible spectrum. It has since been found that any light, unless it is monochromatic, is a mixture of various colors or, better, radiations, since they are not necessarily visible radiations. These radiations differ from one another in wave length. A spectroscope is an instrument which separates these different wave lengths or disperses them into a spectrum for visible observation. The means of dispersion is usually a prism or a diffraction grating.

Prisms give maximum brilliance and increasing dispersion towards the violet end of the spectrum while gratings give ample brilliance and equal dispersion over the entire spectrum.

The diffraction grating is either a flat or curved surface of glass or metal ruled with very fine parallel lines, usually from 15,000 to 30,000 per inch.

In 1883, Rowland invented the concave grating and demonstrated that a simple geometrical relation lay between the slit (through which light from a burning sample passes), the concave grating (which is, in fact, a concave mirror with lines ruled across its surface) and the focusing plane. They lie on the Rowland circle, the diameter of which is equal to the radius of curvature of the concave mirror.

Lens System 2.

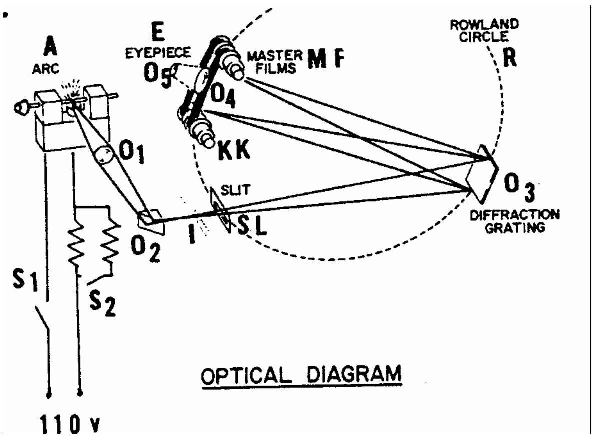
Our instrument uses a concave diffraction grating of half a meter radius of curvature. This was chosen for several reasons, but mainly for compactness, replaceability, evenness of dispersion and reliability. It has 15,000 lines per inch and has been especially developed for Spectrex. (See Appendix V. 2).

Any spectroscope is made up of four basic parts:

a. The light source.
b. The slit (through which the light is passed and hence is narrowed).
c. The means of dispersion.
d. The viewing plate, onto which images of the slit are focused.

These all have definite requirements if accuracy is to be obtained. The light source has to be intense and the power dissipated strong enough to cause the sample under study to emit light. The slit has to be narrow enough to cause a clear image

which is not too faint. The grating should have high resolution and the viewing plate should be so positioned that it is easy to observe.
In the Vreeland spectroscope, the Rowland circle is in a **vertical plane.** In this, it is a unique design and has the advantage of having the eyepiece at the natural level of the eye without making the instrument top heavy. Theoretically, the arc should be just in front of the slit, but in practice, it is found that the arc heat would reach the delicate film and film mechanism. Hence, the arc holder is placed to the side for easy left-hand operation and the light fed to the slit via a gathering lens and right angle prism.



The above diagram illustrates just how a spectrogram of a sample is placed in juxta0position with two continuous spectra by which the standard spectrograms are illuminated.

The lens, 01, is located on the optical axis between the arc and the slit. Its focal length and position with respect to the arc and slit are so determined that it has conjugate foci at the arc and at a point 1, in proximity with the slit. The distance A-01 is purposely made less than the distance 01-1, so that a magnified image is projected.

The concave diffraction grating 03 is located on the optical axis so that its primary focus is at the slit SL, its secondary focus is substantially coincident with the image of the arc at 1 and its diffraction focus curve together with the continuous spectra of the incandescent electrode tips.

If the image of the electrode tips 1 is located at the secondary focus of the grating, the lines separating the line spectrum of the arc from the continuous spectra of the carbon tips will also be sharply defined. The width of the line spectrum between the two continuous spectra is approximately equal to the distance between the images of the electrode tips at 1.

An eyepiece E is employed to view the spectrum and spectrograms. A field lens 04 is located close the spectrum and has a diameter sufficient to embrace a major part of the visible spectrum.

A smaller eye lens 05 ensures that the combined effect give a focal field coincident with the focal surface of the projected spectrum. This is made possible over the complete optical spectrum by mounting the eyepiece on a swinging arm having a slotted end U pivoted at a point near the grating 03.

A spring-loaded pin rides on the guide G and determines the focal position of the eyepiece in coincidence with the focus of the spectrum. Thus, there is no parallax error in comparing the lines of the spectrum with those of the standard spectrograms.

The Master Films and Their Uniqueness 3.

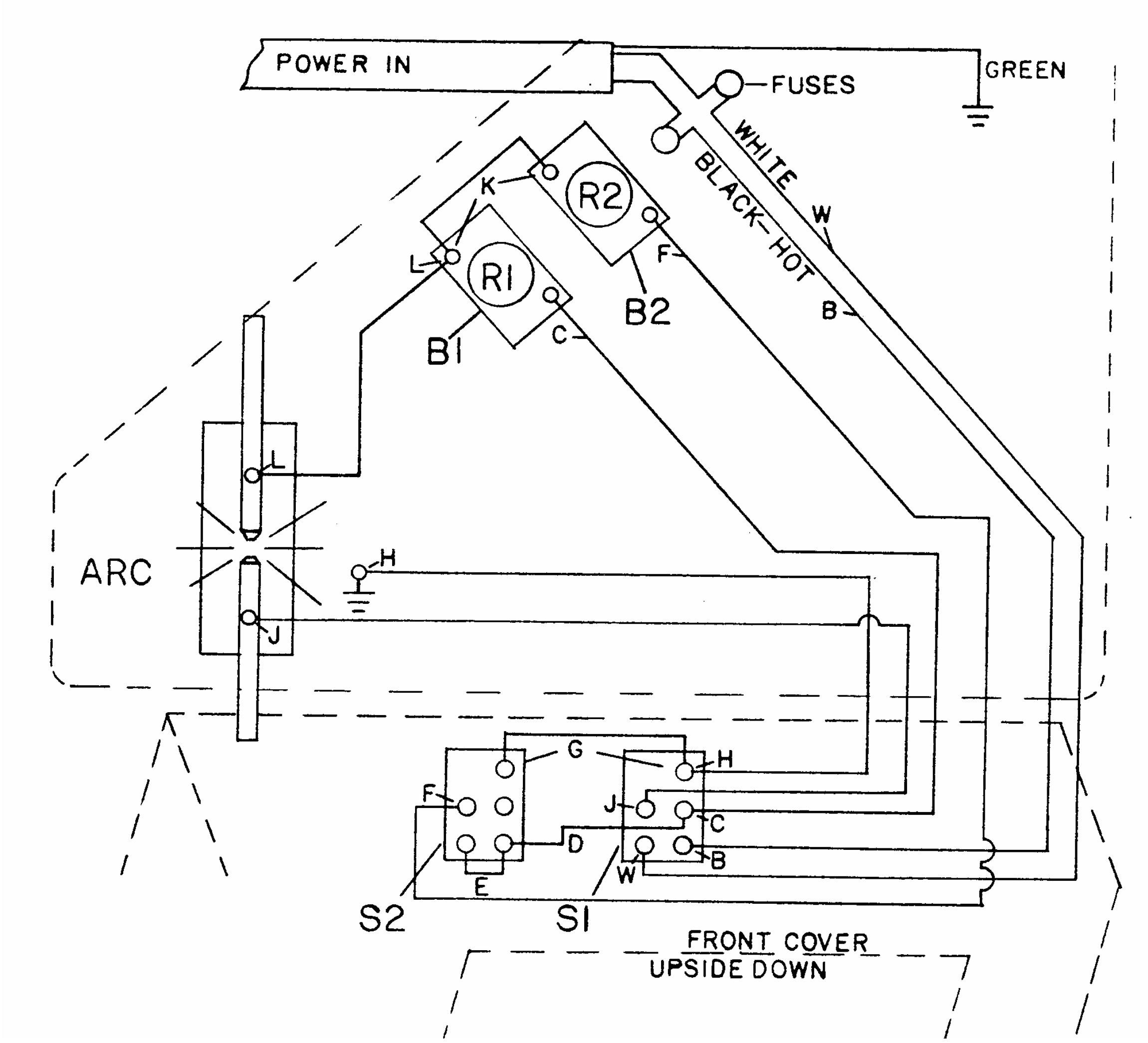
Throughout the years various means have been devised to "unscramble" the many lines which appear on the viewing plane of a spectroscope when a specimen is burnt, for the lines of the different elements are all jumbled together. The cheapest types have an angstrom scale and the wave length of each individual line is laboriously noted and looked up in tables. Not only is this slow but inaccurate. A more sophisticated type has two arc sources. The specimen is burnt on one and a pure sample of one element on the other. The light from both is brought together and the spectral lines compared. This **does** give a definite go/no go basis for analysis **but for only one element at a time.**

The instrument is inevitably expensive, slow in analysis and requires a set of pure samples, which can all too easily be misplaced or contaminated. The most expensive instruments have intricate arrays of photo-electric cells and/or photographic plates and analysis is slow and expensive. They are fine for quantitative work but for quick qualitative analysis the system used in the Vreeland instrument is unique, precise and easy to operate.

The standard spectrograms are recorded as positive on translucent mylar photographic film. Mylar is used for strength and lack of shrinkage. The opaque portions of the film mask the continuous spectra from the carbon arcs, except on their transparent lines which are representative of the spectrum of an element and permit the passage of lines of light corresponding to the lines of the spectrum. Hence, a system is provided whereby simple, direct comparison will indicate whether or not any particular element is present.

The film holder incorporates a special tension control so that each of the master films is controlled by a two directional knob. It is possible to scan each film within 15 seconds.

Electrical Circuit Diagram 1.



S 1 Power on/off switch
S 2 Heat Control switch

R 1 . R 2 Ballast resistors (1000 watt)

Safety

1) A 3-prong socket should always be used. The pilot light is connected between high line and ground. It a 2-prong socket is used, the light and ground. It a 2-prong socket is used, the light will not go on when the power on/off switch is pushed. This is a reminder to ground the instrument **as soon as possible.**

2) Two 15amp fuses are provided on the high and low sides of the "power-in" line.

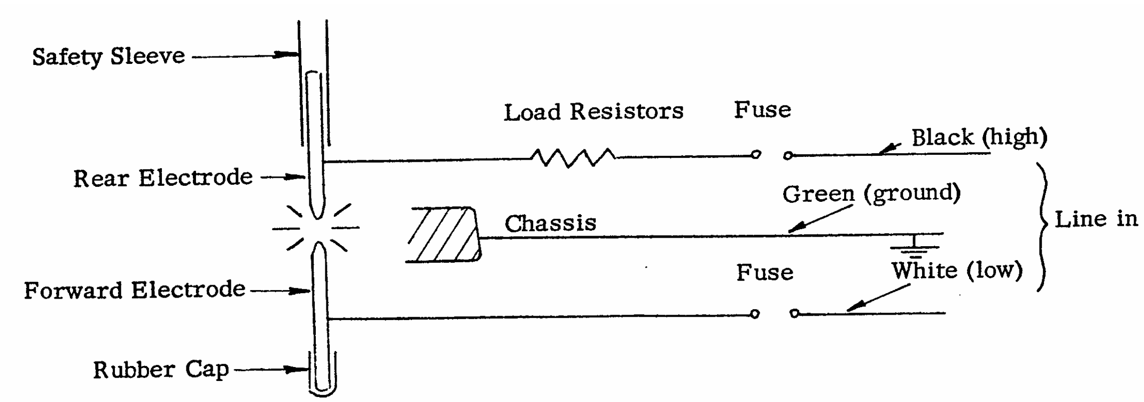
3) S 1 is a double throw switch so that both sides of the arc block are isolated until the instrument is turned on.

4) The load resistors are in the "hot" line so that they would limit current in case of electric shock.

5) The high (black) side of the line is connected to the rear electrode which is protected by the rear safety sleeve.

6) The low (white) side of the line is connected to the forward electrode which is protected by the red, rubber-cap provided with the instrument.

7) The instrument is grounded. (Green Wire).



The reason the low side is not grounded is that there is always the possibility the operator would use a three to two prong adaptor and plug in the instrument to a two-hole socket. In this case, the instrument would not be grounded but **floating.** There would also be the possibility that the two-prong plug would be reversed so that the high line would go to the front electrode. **If this line were connected to the chassis (which is now not grounded) the whole instrument would be at high voltage causing extreme hazard, especially at 220 volts.**

By not connecting the low side to ground, all that would happen is that the forward electrode would be at high voltage; hence, the value of using the red rubber cap. If touched by the operator, the electrode would give a shock but would immediately break off and disconnect itself, avoiding any serious danger.

Spectrex has chosen the lesser of the two evils. If the customer is absolutely sure that the instrument will be grounded at all times, the base cover may be removed with the low side of the line (white) grounded.
If the pilot light does not go on when the instrument is switched on, this is a strong indication that the instrument is not grounded.

We have found that, within the United States, even though wiring codes demand 3-wire grounded outlets, the average home and motel still use 2-wire outlets. Due to the portability of the Vreeland, it is often used in such locations, by prospectors, geologists, chemists and others.

The final alternative is to use an isolation transformer but to carry the current necessary for high heat arcing, it would weigh at least 40 lbs., eliminate the spectroscope's portability, and increase the price. Every customer we have talked to about this matter would prefer the avoidance of the transformer.

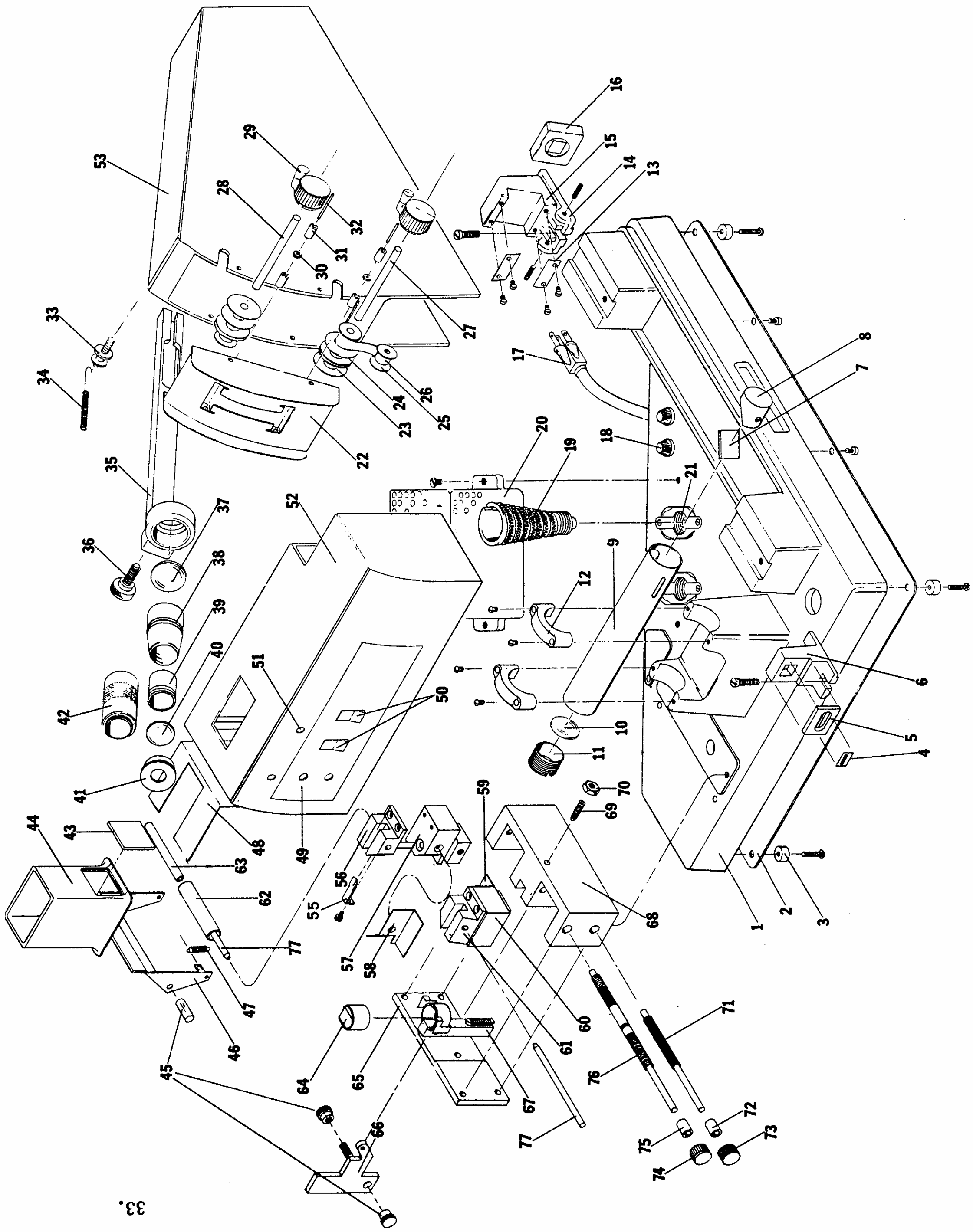
Parts List 2.

Vreeland Spectroscope Model 7
(see exploded assembly drawing)

Code for Exploded View	Catalogue Number	Description
Base		
1	0010000	Base casting
2	0020000	Base cover
3	0030000	Rubber feet
Slit Assembly		
4	0240000	Slit
5	0231000	Light shield
6	0230000	Slit holder
Periscope Assembly		
7	0200000	Front surface mirror
8	0180000	Mirror mount
9	0170000	Periscope tube
10	0190000	Lens
11	0210000	Ring-locking
12	0220000	Clamp-periscope
Grating Mount		
13	0270000	Clamp-grating
14	0250000	Grating adjustment base
15	0260000	Grating mount
16	0280000	Diffraction grating
Power Supply		
17	0060000	Power cable/plug
18	0062000	Fuse holder
19	0100000	Resistor-load

20	0080000	Resistor cover
21	0090000	Socket-resistor
Film Holder Assembly		
22	0290000	Film holder frame
23	0310000	Spool-tension
24	0311100	Spool-non-tension
25	0312000	Spool-negator idler
26	0313000	Negator spring
27	0320200	Rod-spool drive
28	0320100	Rod-spool drive
29	0380000	Knob film holder
30	0360000	Roller spacer
31	0350000	Roller
32	0340000	Roller pin
Eye Piece		
33	0480000	Post-pivot
34	0481000	Spring-arm
35	0410100	Eyepiece arm
36	0462000	Positioning screw
37	0420000	Lens (achromat)
38	0430000	Eyepiece tube
39	0440000	Lens tube
40	0450000	Eye lens
41	0460000	Eye guard
42	0431100	Camera adaptor
Covers & Attachments		
43	0510000	Viewing glass
44	0494000	Chimney
45	0496000	Handle-awning
46	0495000	Awning
47	0497000	Spring-awning
48	0500000	Heat baffle
49	0520000	Control panel
50	0522000	Switch control
51	0523000	Pilot light
	0490000	Pilot light (for 220V)
52	0530000	Front cover
53		Main cover
Arc Block		
55	0118000	Spring electrode tension
56	0112200	Electrode block
57	0111200	Sliding block
58	0116100	Heat protect apron
59	0116200	Heat protect apron
60	0111100	Sliding block

61	0112100	Electrode block
62	0151000	Electrode protection tube
63	0149000	Electrode retractor
64	0120000	Hearth-platform (fixed)
65	0153000	Slide plate
66	0123000	Alignment bar
67	0122000	Hearth holder post
68	0110000	Arc block body
69	0124000	8-32xl set screw-tapered
70	8310000	8-32Hex nut
71	0115000	Pinion shaft
72	0114100	Bushing
73	0141000	Knob – specimen feed
74	0140000	Knob – electrode feed
75	0114200	Bushing
76	0113000	Shaft electrode drive
77	0160000	Electrode
Accessories & Optical Attachments		
77	0160100	Electrode
	0130100	Ceramic hearths
	0129000	Graphite squares
	0135000	Quartz powder 99.999% pure
	0131000	Carbon cups
	0133000	Loading platform
	0134000	Ramrod
	0132000	Loading funnel
	0120000	Fixed hearth with 1/8″ dia. Hole drilled
	0160900	Electrodes drilled with 1/8″ dia. Hole
	0570000	Instruction manual
	0560000	Reference chart (for master films)
	0550000	Tweezers
	0582000	Dust cover
	0431100	Camera adaptor (tube)
42	0551000	Wire brush
	0552100	Wire brush handle
	0162000	Protection sleeve for front electrode (red rubber)
	0583000	Book *Applications for the Vreeland Spectroscope/ Spectrograph* by Prof. Welton J. Crook
	6636000	Video spectra-scan, closed circuit TV attachment
	6630000	*Solarscope* attachment (for viewing solar spectra)
	1150000	Cassette tape/slide presentation *The Vreeland Spectroscope*
	6620000	Digitrex digital camera attachment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