831-914-255-5000
Micromatic optronics Part No 2
Price 180.00

BRICE-PHOENIX

DIFFERENTIAL REFRACTOMETER

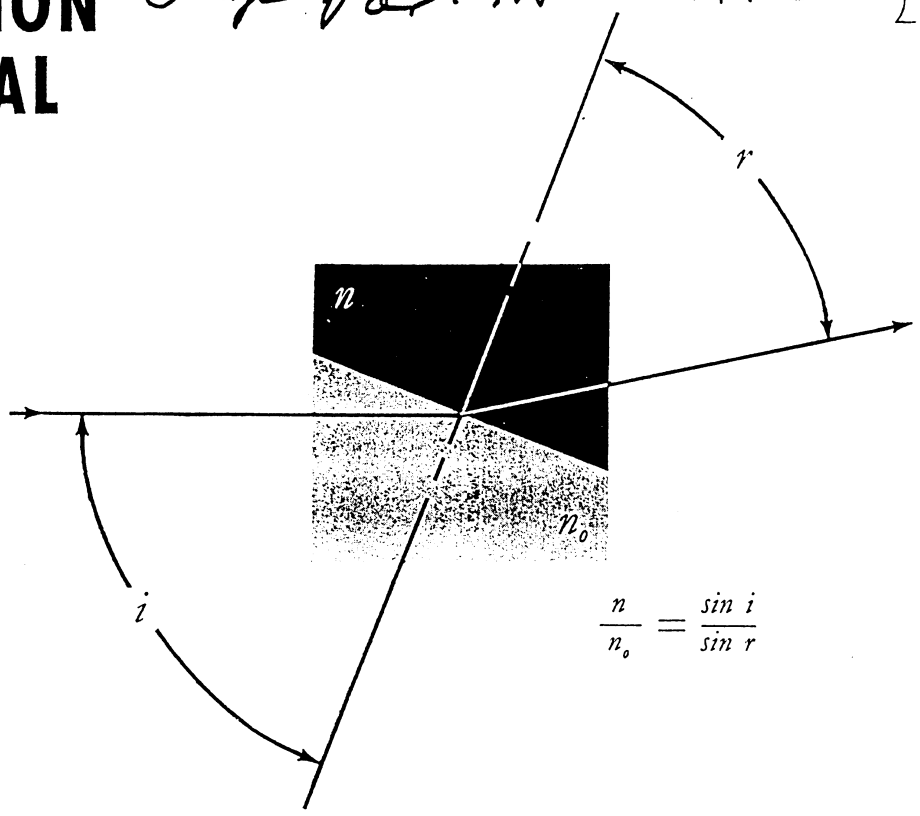
VISUAL LABORATORY TYPE

MODEL BP-2000-V

Gardiner N.Y.
Divoy ~~Dubois~~ Rt 208
Virtis 2525

Longs Part No AH-3 26.00

OPERATION MANUAL



$$\frac{n}{n_0} = \frac{\sin i}{\sin r}$$

PHOENIX PRECISION INSTRUMENT COMPANY
3803-05 NORTH FIFTH STREET
PHILADELPHIA 40, PENNSYLVANIA

MANUAL
BP-2000-V

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OPERATION MANUAL

BRICE-PHOENIX DIFFERENTIAL REFRACTOMETER

I. INTRODUCTION

The Brice-Phoenix, Model BP-2000-V, (formerly B-S) Differential Refractometer was designed for the precise measurement of the *Difference in Refractive Index* between a dilute solution and its solvent (see Brice and Halwer, J. Opt. Soc. Am., 41:1033, 1951). The limiting sensitivity is about 3 units in the sixth decimal place of refractive index difference, while the range is .01 units. A special differential cell, obtainable as an extra-cost accessory, *Phoenix Cat. #R-100-4*, extends the range of the instrument to .07 units and reduces the sensitivity by a factor of one-seventh to approximately 2 units in the fifth decimal place.

Differential instruments are superior to conventional refractometers not only in accuracy but also in simplicity of temperature control. Ambient temperature need not be closely controlled since the temperature coefficient of the difference in refractive index between a solution and its solvent is much smaller than that for the refractive index of solution or solvent alone. It is essential, however, that solution and solvent in the differential cell have the same temperature to 0.01° C or better.

The instrument is particularly suited to the determination of refractive index increments required in the light scattering method of evaluating high molecular weights. However, it also has application in numerous other techniques requiring the measurement of refractive index differences.

II. GENERAL DESCRIPTION

All components are mounted on a rigid, normalized cast aluminum optical bench *(Figure 1, item 1)*, approximately 122 cm long by 13 cm wide. A Mercury Vapor Lamp, type AH-3, operated by a transformer on a 115 volt, 60 cycle line, is used as a light source. The lamp housing *(Figure 1, item 2)* is equipped with a filter turret *(Figure 1, item 3)*, mounting two filters for isolating either the blue *(436 mu)* or green *(546 mu)* wavelengths. Extra openings are provided in the filter turret which allows installation of special filters, if other wavelength isolations are desired.

A semi-transparent mirror *(Figure 1, item 4)*, permanently mounted on the optical bench between the light source and the aperture slit, is fixed at an angle of 45° to the optic axis. This mirror permits the use of either the standard mercury lamp or an accessory light source, which can be mounted at 90° to the optic axis, using the mirror as a reflecting surface.

The monochromatic light beam, after passing through the semi-transparent mirror and a vertical slit *(Figure 1, item 5)* of adjustable width, enters the entrance window of the jacketed cell housing *(Figure 1, item 6)* which is adapted for circulation of constant temperature liquid, and is incident along the normal to the first surface ot the differential refractometer cell. The cell is fabricated of sinter-fused optical glass 15 mm square inside with plane parallel windows, a removable cover, and a thin (1.2 mm) diagonal glass partition which divides the cell into two equal compartments, one for solvent and one for solution. The cell holder inside the housing can be rotated about a vertical axis through 180° by means of a handle projecting from the fixed housing. Rotational stops are provided by Allen-head screws. The glass cell is clamped symmetrically in its holder by set screws so that the axis of rotation passes through the center of the cell and its polished faces are perpendicular to the optic axis.

The standard differential refractometer cell, normally supplied with the Brice-Phoenix Differential Refractometer, has two opposing faces polished so that the light beam forms an angle of incidence of approximately 69° with the normal to the surface of the partition. This cell is ideal for refractive index measurement up to 1.62 and for determination of refractive index difference up to about 0.01 units. For refractive index measurements above 1.62, a special differential cell having all four faces polished must be ordered. This special cell, *Phoenix Cat. #R-100-4*, may be used in the low sensitivity position in which the angle of incidence at the partition is approximately 21°. The sensitivity is reduced by a factor of one-seventh, but the range is extended seven times to approximately 0.07 units. Both standard and special differential cells which are adopted for use with volatile liquids, are available. The modifications include a cover plate permanently fused to the cell and supplied with two removable ground glass penny-head stoppers for filling. All of the aforementioned differential cells, on special order, can be fabricated from ultraviolet transmitting quartz glass. (Refer to Accessories and Parts List.)

The light beam, emerging from the differential refractometer cell, has been deviated through an angle which is proportional to the refractive index difference between the two liquid media, and leaves the cell housing through the exit window. A projector lens *(Figure 1, item 7)* projects the image of the slit onto the focal plane of the objective of a microscope *(Figure 1, item 8)* fitted with a filar micrometer eyepiece having a 10 mm fixed scale *(Figure 1, item 13)* and a drum *(Figure 1, item 12)* divided to 0.01 mm. The iris diaphragm of the projector lens *(item 7)* is useful in controlling the brightness of the slit image; relatively small apertures will favor absence of annoying multiple slit images. The microscope *(Figure 1, item 9)* is movable along its longitudinal axis allowing focal adjustment of the slit image.

A cross hair within the eyepiece is moved, by rotation of the divided drum, until it superimposes the slit image, enabling the eyepiece scale to be read to four significant figures.

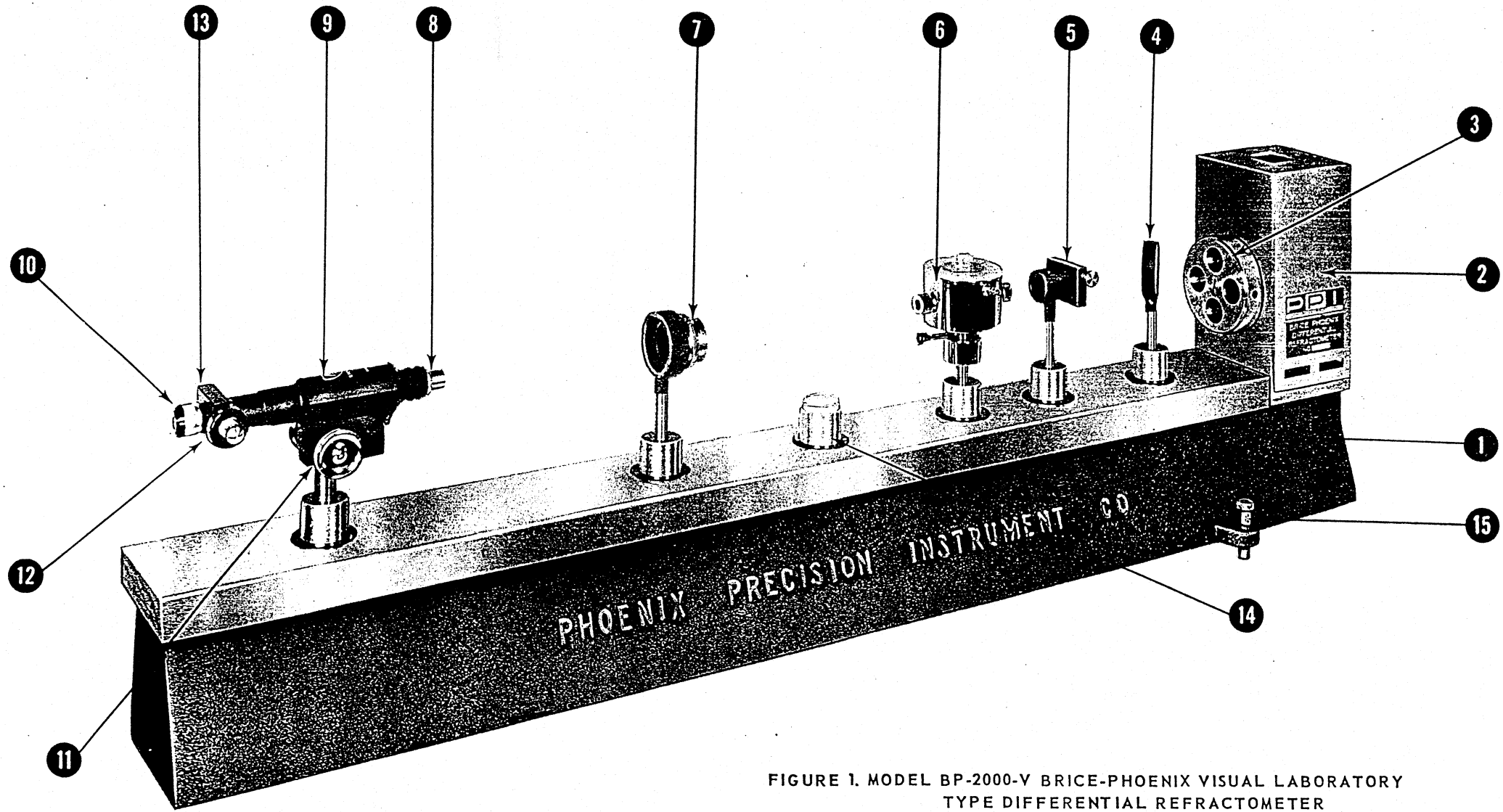
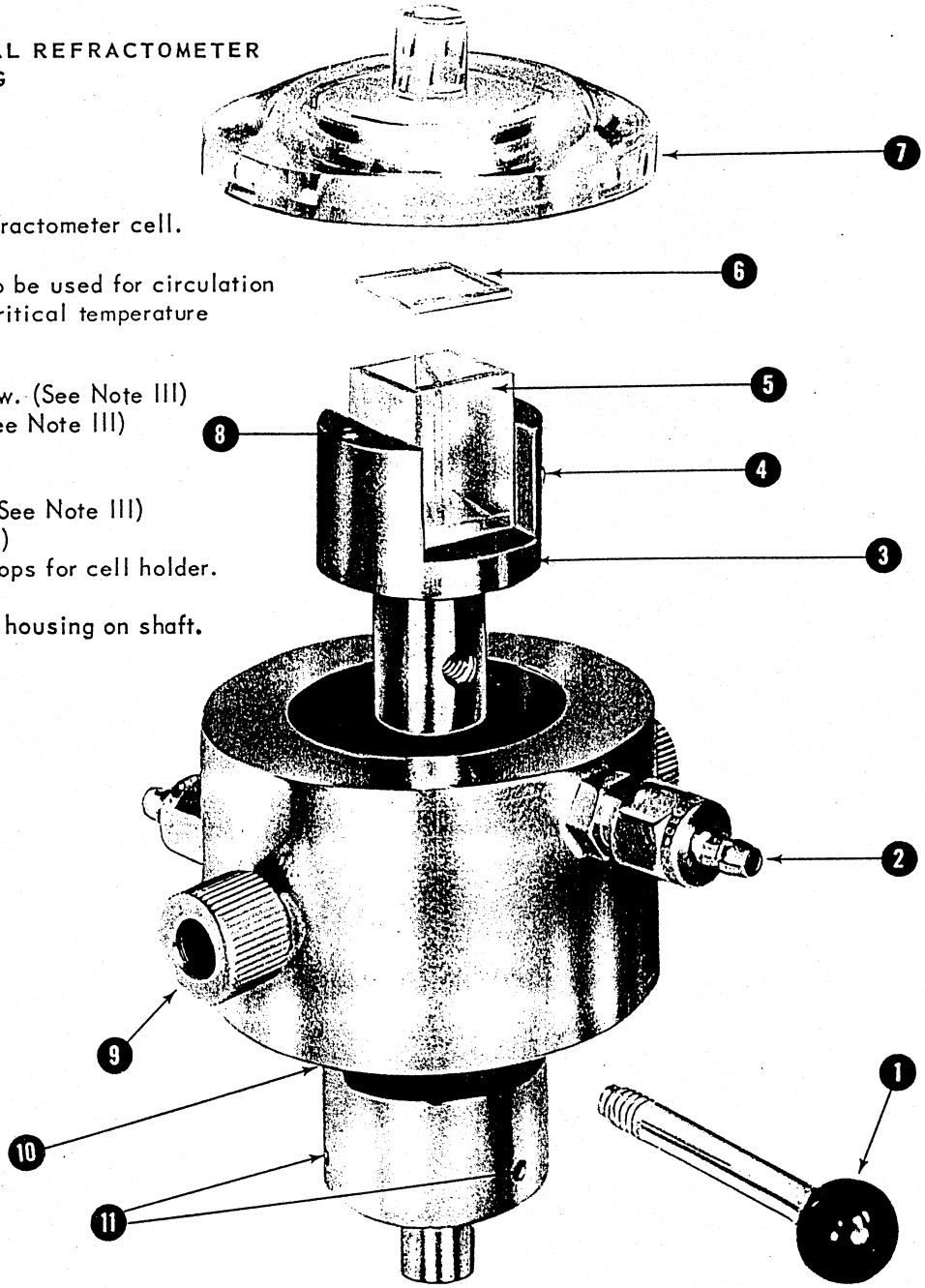


FIGURE 1. MODEL BP-2000-V BRICE-PHOENIX VISUAL LABORATORY TYPE DIFFERENTIAL REFRACTOMETER

1. Optical Bench
2. Lamp Housing (The lamp cover may be removed by sliding it up within its tracks.)
3. Filter turret
4. Semi-transparent mirror
5. Adjustable slit
6. Jacketed cell housing
7. Projector lens
8. Microscope objective
9. Microscope
10. Eyepiece focus
11. Image focal adjust
12. Filar micrometer drum
13. Filar micrometer eyepiece
14. Spirit level
15. Leveling bolt

FIGURE 2
EXPLODED VIEW OF DIFFERENTIAL REFRACTOMETER CELL HOUSING



1. Handle for rotating differential refractometer cell. (See Note I)
2. Connection for 1/8" I.D. tubing to be used for circulation of constant temperature fluid, if critical temperature control is required. (See Note II)
3. Cell holder. (See Notes I and III)
4. Cell Alignment adjusting set screw. (See Note III)
5. Differential refractometer cell. (See Note III)
6. Cell cover plate.
7. Cell housing lid.
8. Cell position locking set screw. (See Note III)
9. Window retaining nut. (See Note II)
10. Set screws providing rotational stops for cell holder. (See Note III)
11. Set screws fixing position of cell housing on shaft. (See Note III)

NOTE I
When it is desired to remove the cell holder from the cell housing, unscrew the handle from the shaft of the cell holder. The cell holder assembly may now be lifted up and out, touching only the ground glass sides of the cell.

NOTE II
Plane glass windows are available (see Accessories List) and must be installed when temperature control is required.

NOTE III
These set screws must not be tampered with. The differential refractometer cell must not be removed from the cell holder, except for replacement. Alterations in the cell position within its holder, or in the position of the rotational stops will require that a difficult realignment procedure be carried out by skilled personnel.

III. OPERATING INSTRUCTIONS

1. Turn on the light source lamp and allow approximately fifteen minutes for stabilization.

2. Rotate the filter turret to select the proper filter for the desired wavelength isolation.

3. Flush both compartments of the differential cell *(Figure 2)* several times with the solvent to be used. Use a 1 ml pipette for filling, and a hypodermic syringe, with needle end ground flat, for emptying the compartments. When filling and withdrawing, avoid spilling fluid on the outer faces of the cell. Strips of clean ink blotting paper, cut to measure about 1/8" by 2", are convenient aids in the final removal of traces of fluid remaining after rinsing.

4. Fill both compartments of the differential cell with the solvent to be used. One milliliter of liquid in each compartment is sufficient. Place the glass cover on the cell and the plastic cover on the jacketed cell housing.

5. Turn the handle of the rotatable cell holder against the stop so that it points toward the lamp. Readings with the cell in this position will have the subscript 1.

6. Focus the microscope on the narrow slit image. The width of the vertical slit should be adjusted to the minimum dimension compatible with sharp definition of the image. The iris diaphragm of the projector lens generally remains at f:11, but the aperture size may be increased or decreased as desired to vary the brightness of the image.

7. By means of the micrometer drum, carefully set the cross hair, within the eyepiece, so that it is aligned with the center of the slit image. The dark background of the fixed eyepiece scale may be illuminated sufficiently for scale reading by holding a white card at a slight angle in front of the microscope objective, so that room light is reflected into the microscope. Read d_1' from the eyepiece scale and the micrometer drum. Displace the cross-hair, re-align it, and read d_1' again. Repeat several times, recording all readings.

8. Turn the handle of the differential cell holder through 180° (handle now points toward observer), refocus the microscope, and repeat the operations outlined in step #7. With the cell in this position, however, the readings are to be recorded as d_2'.

9. Calculate the solvent zero reading by subtracting the average d_1' reading from the average d_2' reading. The difference may be positive or negative. To illustrate with readings typical of distilled water solvent:

Readings with cell holder handle pointing toward lamp -

(1)	4.997
(2)	4.995
(3)	4.998
(4)	4.997
(5)	4.998

$d_1' = 24.985 \div 5 = 4.997$ (average)

Readings with cell holder handle pointing toward observer

(1)	5.003
(2)	5.005
(3)	5.001
(4)	5.002
(5)	5.004

$d_2' = 25.015 \div 5 = 5.003$ (average)

Solvent Zero Reading $(d_2' - d_1') = 5.003 - 4.997 = +0.006$

It must be remembered that this zero reading refers only to the given solvent, using a specified wavelength. The instrument was aligned at the factory so that d_1' and d_2' are near 5 (mid-scale) on the eyepiece scale, and so that $d_2' - d_1'$ is very small for distilled water.

IV. CALIBRATION

The calibration of the instrument is accomplished by using, as a reference, a solution having a known refractive index difference between solution and solvent. In the past, a pure sucrose solution in distilled water was used as the reference. Recently, however, we have found a distilled water solution of potassium chloride was more readily reproducible and now use this solution exclusively at our factory. Test solutions for calibration may be obtained from Phoenix Precision Instrument Co. (Prices on request).

In preparing your own test solutions, use potassium chloride, re-agent grade, dried at approximately 90° C for a reasonable period. It is important that some of the distilled water used in preparing the solutions be retained for use as the reference solvent. The distilled water for all calibration solutions must be taken from the same batch since the instrument might detect differences between water from different suppliers or even between various batches. Refer to *Table 1* in selecting and preparing a calibration solution. It is accepted best practice to select a concentration in which the refractive index difference between the solution and its solvent is similar in magnitude to whose which are later to be measured.

If it should be more convenient to use a calibration solution other than potassium chloride, refer to

Tables 2 through *4* for selection of different solutions in a wide range of refractive index differences.

After preparation, the solutions should be placed in new bottles that were rinsed three times with distilled water and inverted for overnight drying.

The following procedure must be followed to calibrate the instrument:

1. Withdraw all solvent from the solution compartment of the differential cell. *(The solution compartment is the one nearest the observer when the cell holder handle is pointing toward the lamp).* Remove all final traces of solvent with strips of clean blotting paper and flush the compartment three times with the calibration solution of known refractive index difference.

2. Fill the compartment with 1 ml of the calibration solution, cover the cell and the cell housing, with the glass or plastic cover provided, and allow approximately five to ten minutes for temperature equilibrium of the solution and its solvent within the cell.

3. Turn the cell holder handle against the zero stop so that the handle points toward the lamp housing. This is the position for reading d_1 of the solution.

4. Without changing the width of the vertical slit, repeat steps No. 6, 7 and 8 listed in Section III, Operating Instructions, and record the readings for d_1 and d_2 of the solution.

5. Determine the solution $d_2 - d_1$ by subtracting the average reading for d_1 from the average reading for d_2.

6. The reading of total displacement, Δ d, corrected for the solvent zero reading, is calculated as follows:

$$\Delta d = (d_2 - d_1) - (d_2' - d_1')$$

where $(d_2 - d_1)$ is the reading of the solution and $(d_2' - d_1')$ is the zero reading of the solvent.

7. Refractive index difference is given by the following equation:

$$\Delta n = k \Delta d$$

where Δ n is the refractive index difference between a solution and its solvent, k is the calibration constant for the selected wavelength, and Δd is the total slit image displacement (solvent zero corrected) in instrument units. Since Δn is a known quantity, and Δd has been determined by measurement the equation can be solved for k.

Once the calibration constant has been determined, any deviation reading of light of the selected wavelength may be directly converted to refractive index difference.

Since the k value for a given wavelength is inversely proportional to the magnification, at that wavelength, of the optical system of the instrument, the formula stated below may be used for calculating the calibration constant for other wavelengths:

$$\frac{k_{436}}{k_{546}} = \frac{M_{546}}{M_{436}}$$

where M_{546} and M_{436} are the magnifications of the optical system at 546 mu and 436 mu respectively.

The magnification factors for your instrument #2199 are:

M at 436 mu = 1.553
M at 546 mu = 1.554
M at 589 mu = 1.558

V. MAINTENANCE

A. ALIGNMENT

The apparatus is properly aligned when: (a) light passes centrally through the slit, cell housing, and along the axis of the microscope, both with and without the lenses and with the cell (filled with a solvent) in the path of the beam: (b) the axis of rotation of the cell passes through the center of the cell; (c) the light beam is incident normally on the cell face for both 0° and 180° positions; (d) with the cell filled with water, the slit image falls near the center of the fixed micrometer eyepiece scale, and there is no appreciable displacement of the slit image for 0° and 180° settings of the cell; and (e) with a test solution (e.g., 0.4M potassium chloride in distilled water) in one compartment and solvent in the other compartment of the cell, the slit images for 0° and 180° positions of the cell are of approximately equal brightness, and show uniform lateral spreading of each slit image on focusing the microscope in and out. This alignment has been attained at the factory and all adjustments have been securely locked. It should not be necessary to alter these adjustments except possibly after replacing a burned-out lamp or when obvious accidental misalignment has occurred.

B. REPLACING MERCURY VAPOR LAMP

Occasional replacement of a defective AH-3 mercury vapor lamp is the only procedure affecting the optical alignment of the instrument which would normally be carried out by the user. The instructions below should be carefully followed.

1. Disconnect the power cable from the electrical outlet.

2. Remove the cover of the light source housing by sliding it up along its tracks.

3. Remove the defective lamp and replace it with a new one, screwing it firmly into the lamp socket.

4. Clean the glass envelope of the lamp with a cloth moistened with alcohol, and wipe dry.

5. Holding the lamp envelope with a clean, dry cloth only, adjust the lamp within its socket so that the capillary is vertical. All subsequent adjustments should be made by handling the porcelain lamp base and not the glass envelope.

6. Loosen the Allen-head set screw on the flange below the lamp base plate, and rotate the lamp base so that the upper, horizontal supporting brackets for the capillary, within the glass envelope of the lamp, are parallel to the optical bench. This must be done so that the front of the capillary is not obscured by the supporting structures. At the same time, vertically align the lamp so that the capillary is centered within the aperture in the front plate of the light source housing. When the proper position is attained, tighten the set screw.

7. The lamp capillary must also be laterally centered within the aperture in the front plate of the light source housing. Loosen the two screws which mount the porcelain lamp socket to the base plate, to permit lateral movement of the lamp. Connect the power cable to the electrical outlet and strike the lamp. Remove the window retaining nut (Figure 2, item 9) from the entrance port of the cell housing. Wait several minutes for the lamp intensity to stabilize. Laterally adjust the position of the lamp base so that the slit of light enters the entrance port of the cell housing precisely at the center of the bore diameter. This is most conveniently done in a darkened room, holding a millimeter scale horizontally at the outer face of the entrance port. When the proper lamp position is attained, tighten the lamp base mounting screws. Recheck the position of the light beam to assure that the adjustment was not altered while the screws were tightened.

CAUTION: *Do not* alter the position of the lamp housing base plate by loosening the three screw mounting the lamp housing to the optical bench.

8. Replace the light source housing cover.

C. CHECK THE CALIBRATION of the instrument occasionally.

D. DO NOT leave acid cleaning solution, concentrated acids, or strong bases in the differential cell for prolonged periods. Clean only with mild cleaning agents when necessary.

E. WHEN THOROUGH CLEANING of the cell becomes necessary, ***do not release the cell from its holder.*** Remove the cell, while still mounted in its assembly, by unscrewing the 1/4" diameter shaft of the cell holder handle and lifting the entire assembly from the cell housing (***Figure 2***). After cleaning the cell without removing it from its holder, replace the assembly and handle.

F. THE USE OF HIGHLY VOLATILE SOLVENTS may present difficulties due to capillary transfer of liquid from one cell compartment to the other between the cell top and its lid, resulting in Δd values which drift with time. Errors in such cases may be minimized by raising the lid slightly above the cell body with aluminum foil spacers and by circulating water at a constant, reduced temperature in the cell housing. Readings on such solutions should be taken without unnecessary delay. A special closed cell, (***see Parts List***), modified for use with highly volatile solvents, may be obtained from PPI.

TABLE 1

REFRACTIVE INDEX DIFFERENCES (Δn) BETWEEN POTASSIUM CHLORIDE SOLUTIONS AND DISTILLED WATER

Reagent grade, dried in vacuum oven at approximately 80 to 100°C
(Calculated from A. Kruis, Ziet. Phys. Chem. 34B, 13, 1936)

Solution Number	Concentration (1) g/100 ml.	Concentration (2) g/100g.H_2O	$\Delta n \times 10^6$ at 25°C 589 mu	578 mu	546 mu	436 mu
1	0.0696	0.0699	96	96	96	100
2	.1067	.1070	146	147	148	153
3	.2799	.2812	382	383	386	399
4	.5964	.5994	810	812	817	845
5	1.0794	1.0869	1457	1460	1469	1521
6	1.4911	1.5037	2005	2009	2022	2093
7	2.9821	3.0250	3962	3969	3994	4135
8	3.9969	4.0703	5271	5281	5314	5500
9	4.4732	4.5647	5879	5890	5926	6136
10	5.9642	6.1217	7766	7781	7828	8105
11	6.4680	6.6526	8398	8414	8465	8763

Concentration: (1) Grams of salt per 100 ml. of distilled water, 25°C.
Concentration: (2) Grams of salt added to 100 grams of distilled water.
NOTE: See general instructions for preparation of test solutions on page 6.

TABLE 2

REFRACTIVE INDEX DIFFERENCES (Δn) BETWEEN SODIUM CHLORIDE SOLUTIONS AND DISTILLED WATER

Reagent grade, dried in vacuum oven at approximately 80 to 100°C
(Calculated from A. Kruis, Zeit. Phys. Chem. 34B, 13 (1936)

Solution Number	Concentration (1) g/100 ml.	Concentration (2) g/100g.H_2O	$\Delta n \times 10^6$ at 25°C 589 mu	578 mu	546 mu	436 mu
1A	0.0938	0.0941	165	165	166	173
2A	.1034	.1037	182	182	184	190
3A	.3362	.3375	587	588	592	614
4A	.5602	.5627	974	976	982	1019
5A	.6866	.6900	1191	1194	1202	1246
6A	.9090	.9142	1572	1576	1586	1645
7A	1.1240	1.1311	1939	1943	1956	2028
8A	1.6465	1.6595	2824	2830	2848	2954
9A	2.0327	2.0513	3474	3481	3504	3634
10A	3.7307	3.7854	6293	6306	6347	6583
11A	6.7405	6.9092	11151	11174	11247	11670
12A	10.488	10.895	16996	17030	17144	17800
13A	16.011	16.988	25260	25313	25480	26430

Concentration: (1) Grams of salt per 100 ml. of distilled water, 25°C.
Concentration: (2) Grams of salt added to 100 grams of distilled water.
NOTE: See general instructions for preparation of test solutions on page 6.

TABLE 3

AMMONIUM NITRATE (NH_4NO_3)

Reagent grade, dried in vacuum oven at approximately 80° to 100°C

Solution Number	Concentration (1) g/100 ml.H_2O	Concentration (2) g/100g.H_2O	$\Delta n \times 10^6$ at 25°C 546 mu	436 mu
14A	37.076	48.340	42420	44540

Concentration (1) Grams of salt per 100 ml of distilled water at 25° C.
Concentration (2) Grams of salt added to 100 grams of distilled water.

TABLE 4

REFRACTIVE INDEX DIFFERENCES (Δn) BETWEEN SUCROSE SOLUTIONS AND DISTILLED WATER

Pure, dried in vacuum oven at approximately 90°C. (Calculated from C. A. Browne and F. W. Zerban, Physical and Chemical Methods of Sugar Analysis, Third Edition, Wiley, New York, 1941 (Table 6, page 1206.)

Percent Sucrose by Weight (g/100g.H_2O)	Refractive Index Difference, $n - n_o$, for 589 mu 20°C.	28° C.
2.000	0.00289	0.00287
4.000	0.00581	0.00576
5.000	0.00728	0.00722
6.000	0.00877	0.00870

NOTE: See general instructions for preparation of test solutions on page 6

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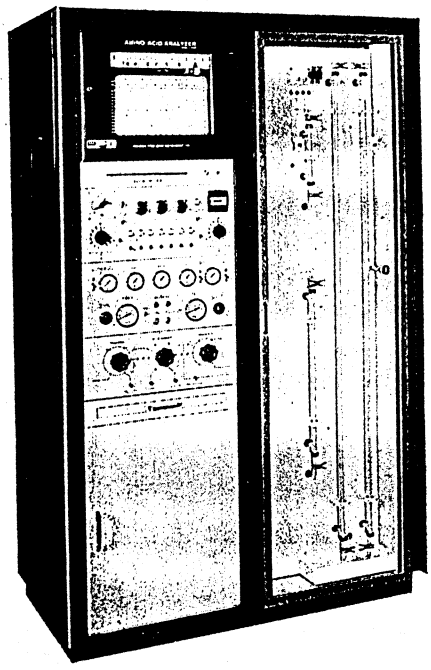
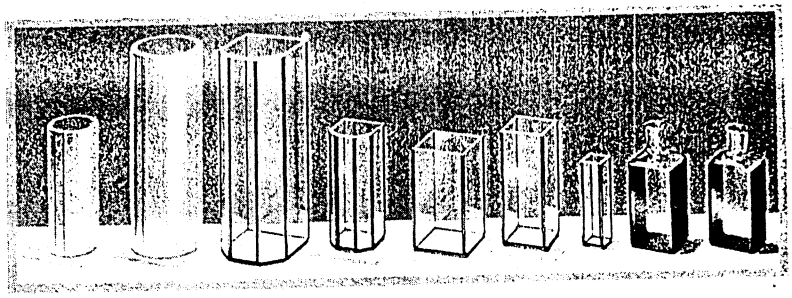
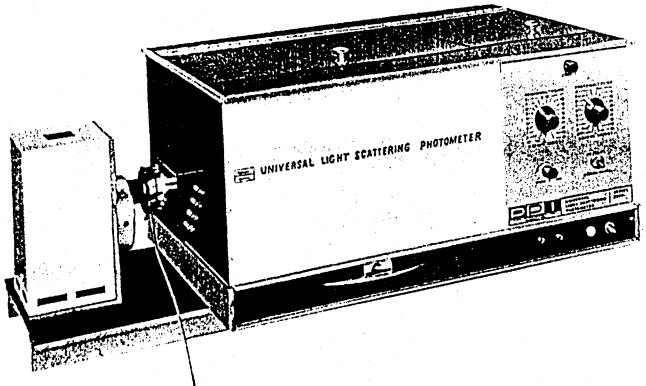
Recording Differential Refractometers for Process Streams
Nephelometers
Continuous Recording Nephelometers



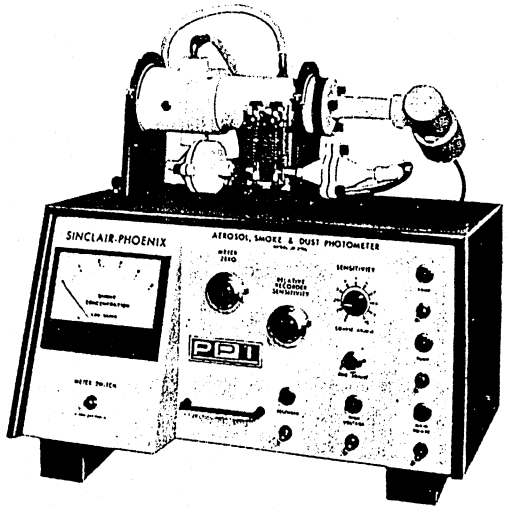
AMINO ACID ANALYZERS



ABSORPTION CELLS



BRICE-PHOENIX LIGHT SCATTERING PHOTOMETERS



AIR POLLUTION PHOTOMETERS

PPI

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Figure 1

0.1283

$dn/dc = (2.479 \times 10^{3} = 2499 \times 10^{1}) \lambda^{-2} + [\text{redacted}] \pm 9.361 \times 10^{-5}$

$r = 0.99989$

KCl/H_2O Hugelin

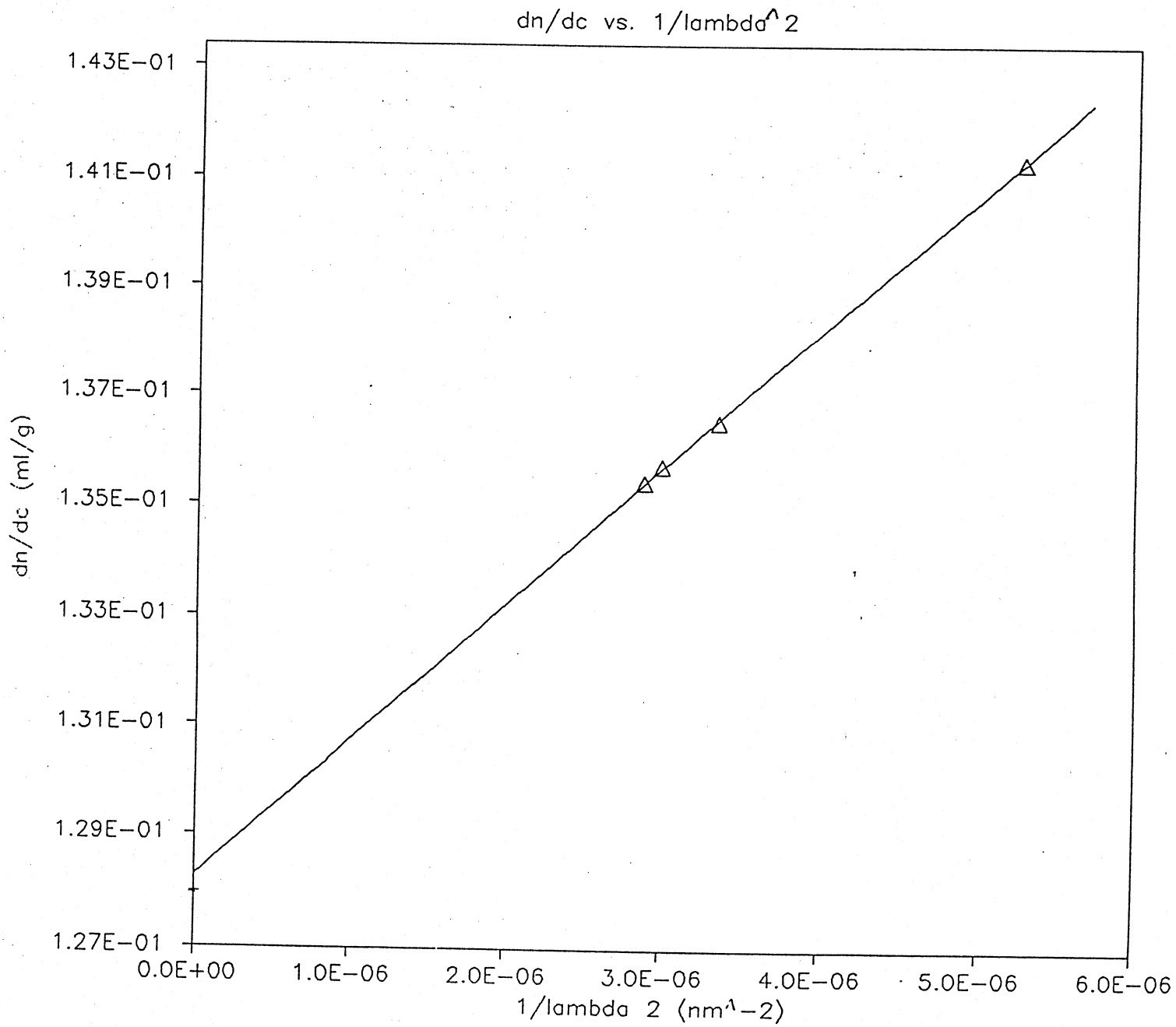

Table 1

λ (Å)	dn/dc (ml/g)
6328	0.13409
5890	0.13525
5145	0.13727
4880	0.13831

off slightly. Should really recalculate.

$\frac{\Delta d}{\Delta c}$ $\left(\frac{\Delta n}{\Delta d}\right)$

$\Delta n = k \Delta d$

Δd / c

$\Delta d = B$

Tahir Jamil Rework of dw/dc 12/19/90

Plotted $\triangle$N Vs concentration of KCl data given in Table #1 at different wavelengths. From the slope of the plot, (dn/dc) (ml/gm) at a given wavelength was determined. These (dn/dc) values were plotted against $1/\text{lambda}^2$(nm). A linear fit to the plot gave a straight line equation from which (dn/dc) at other wavelengths were determined. Two set of data has been used. In the first all KCl data was utilized. The best fit equation is:

$$(dn/dc) = (2390.631436 \pm 26.972628).\ 1/\text{lambda}^2 + (0.1230718 \pm 0.0001011)$$

In the second set of data, only first seven concentrations were used. The best fit equation is:

$$(dn/dc) = (2450.919061 \pm 23.384570).\ 1/\text{lambda}^2 + (0.1257026 \pm 0.0000877)$$

The (dn/dc) values calculated from these two equations at other wavelengths along with Mark deLong's values are:

Wavelength(nm)	Ist Eq.	Second Eq.	deLong
632.8	0.12904	0.13182	0.13409
589.0	0.12996	0.13277	0.13505
514.5	0.13210	0.13496	0.13727
488.0	0.13311	0.13599	0.13831

Similar calculations were performed on NaCl data given in Table #2. The best fit line has the equation:

$$(dn/dc) = (3319.324072 \pm 32.448574).\ 1/\text{lambda}^2 + (0.161810 \pm 0.000122)$$

The (dn/dc) values calculated at different wavelengths are:

Wavelength (nm)	dn/dc (ml/gm)
632.8	0.17010
589.0	0.17138
514.5	0.17435
488.0	0.17575