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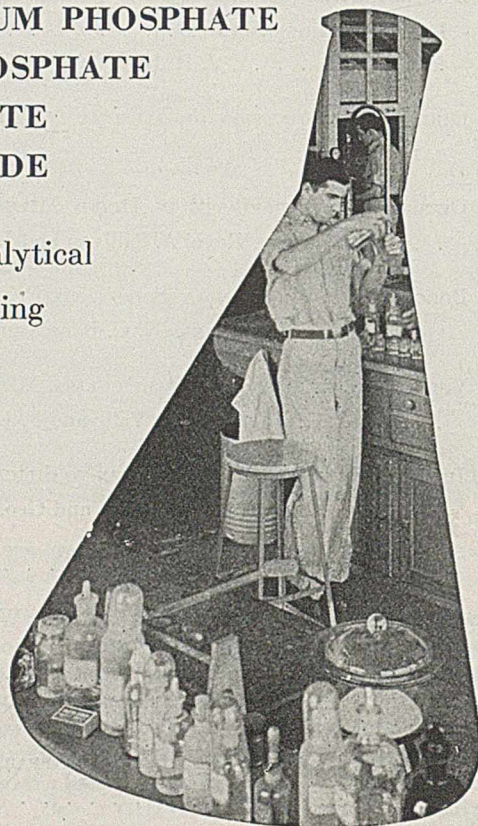
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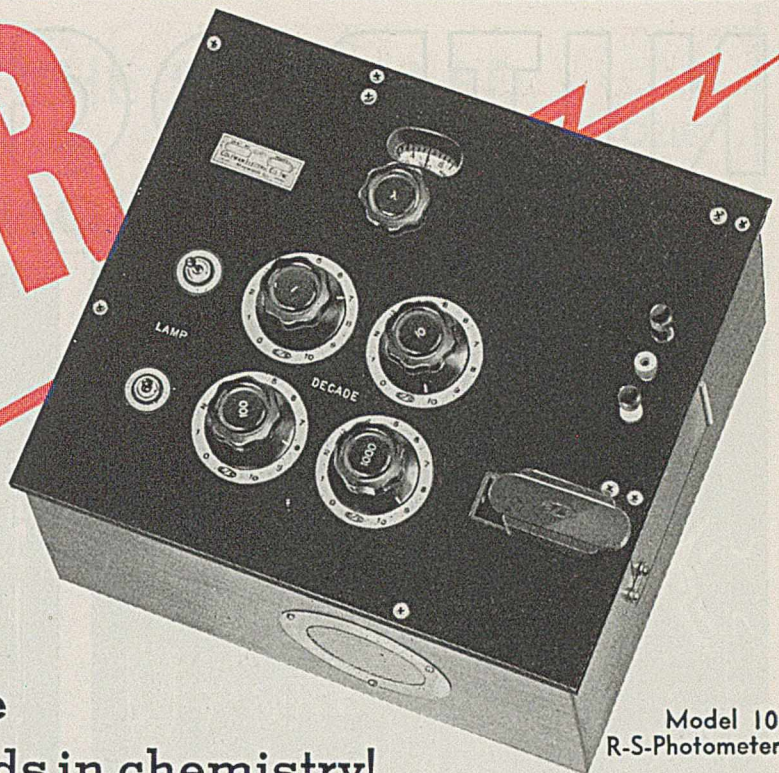
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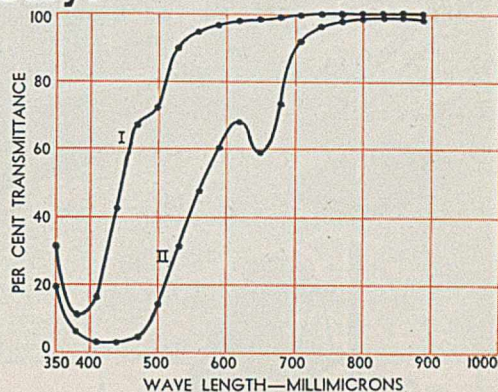
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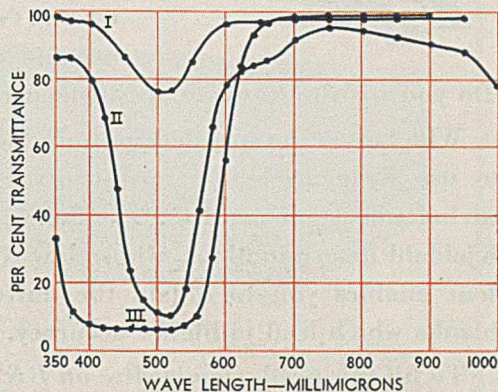
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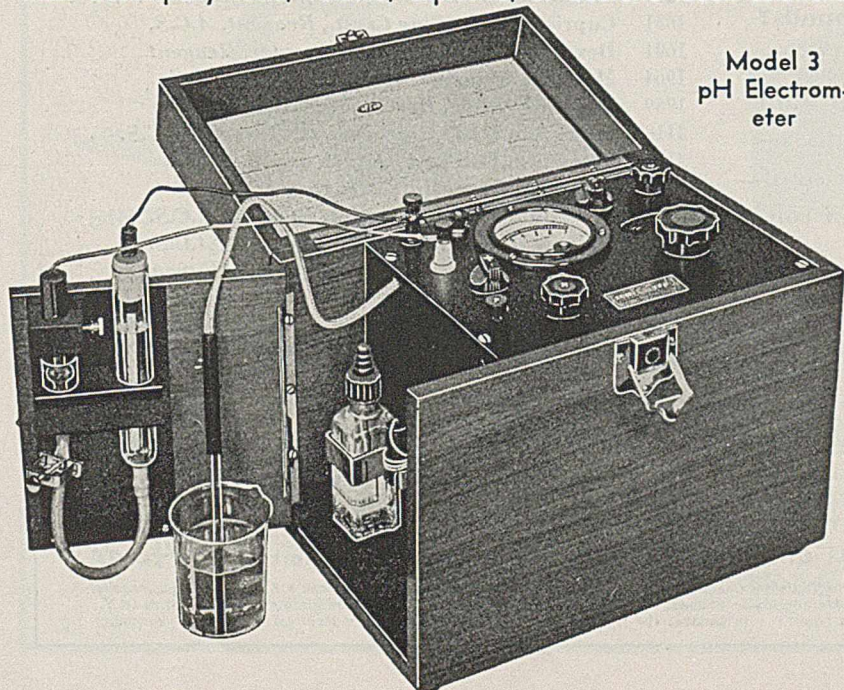
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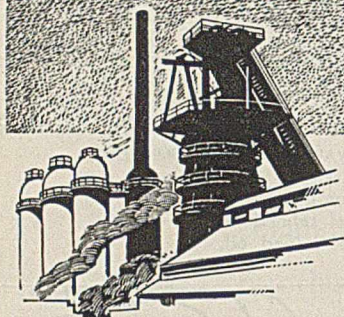
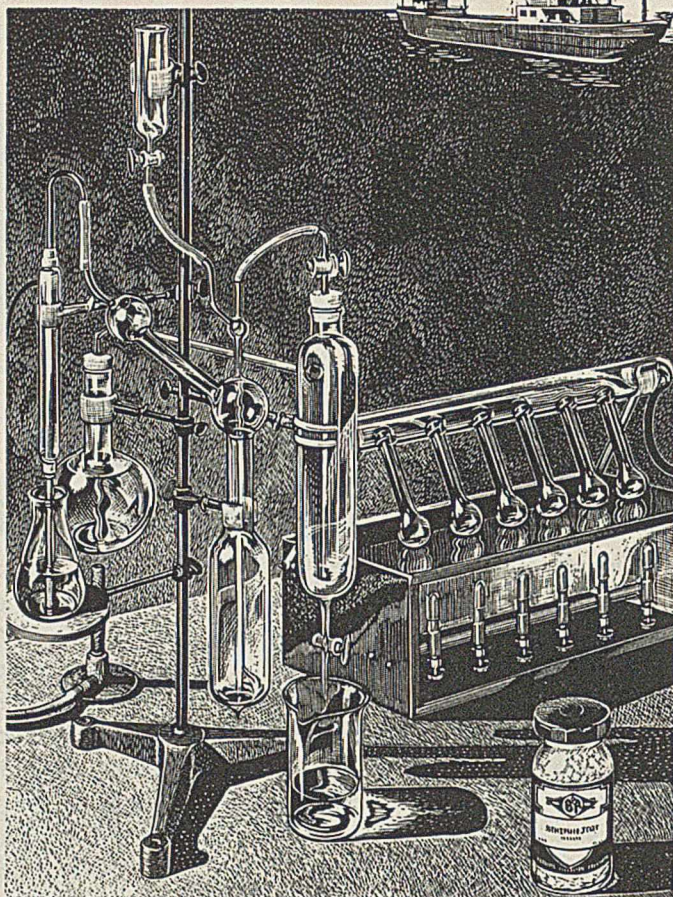
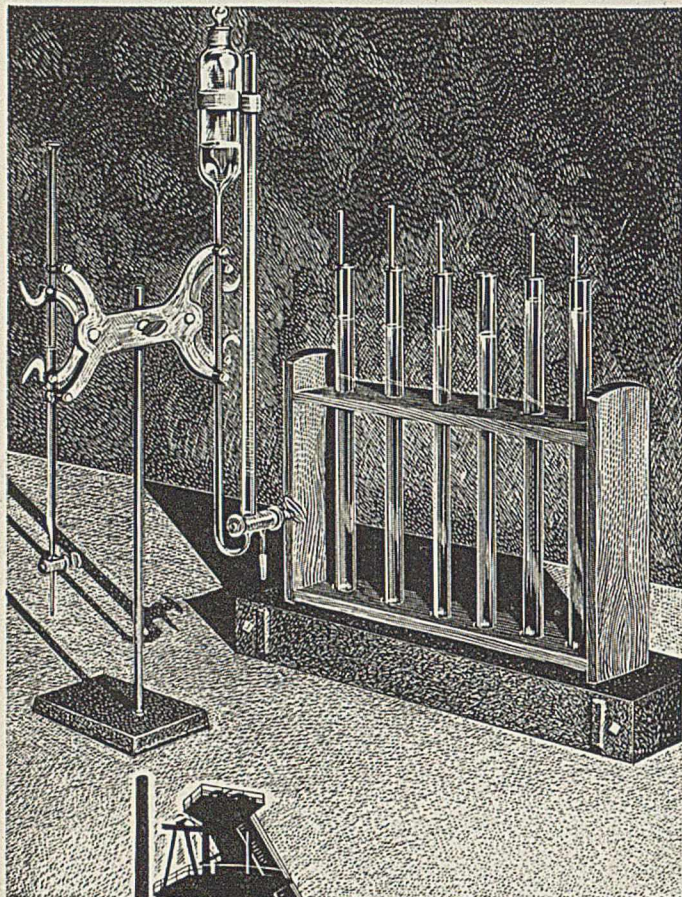
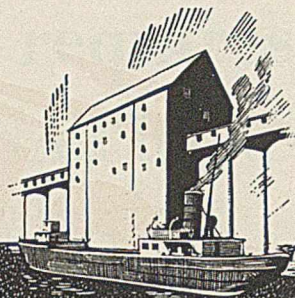
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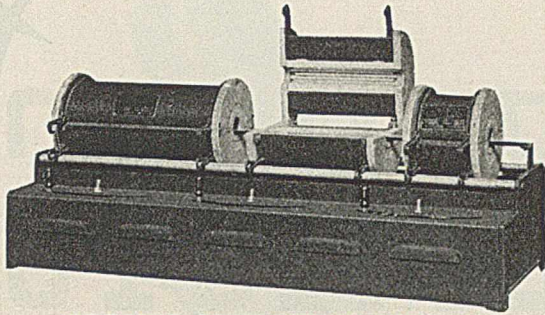
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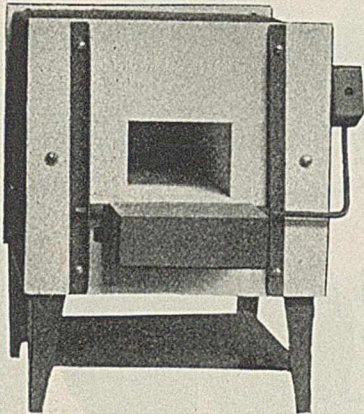
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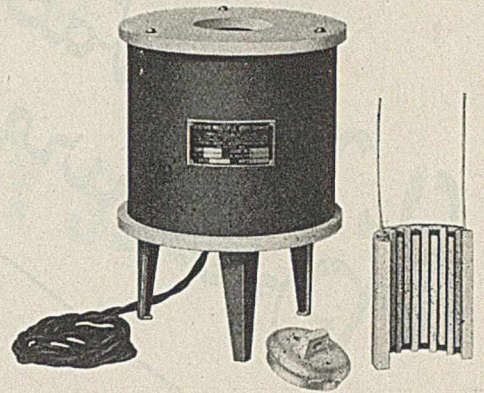
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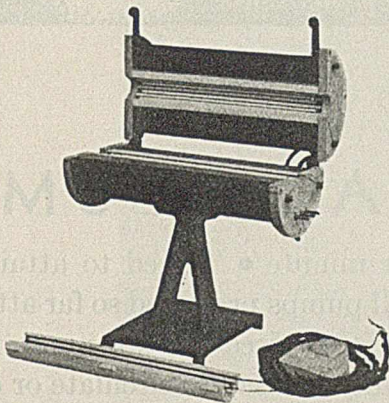
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4 STANDARD TYPES



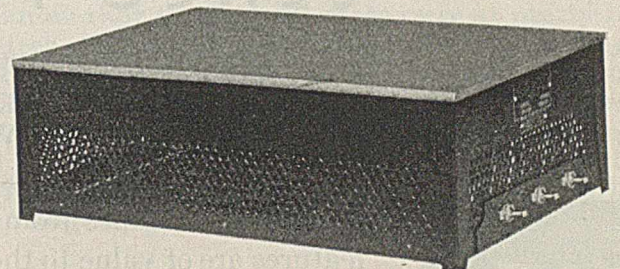
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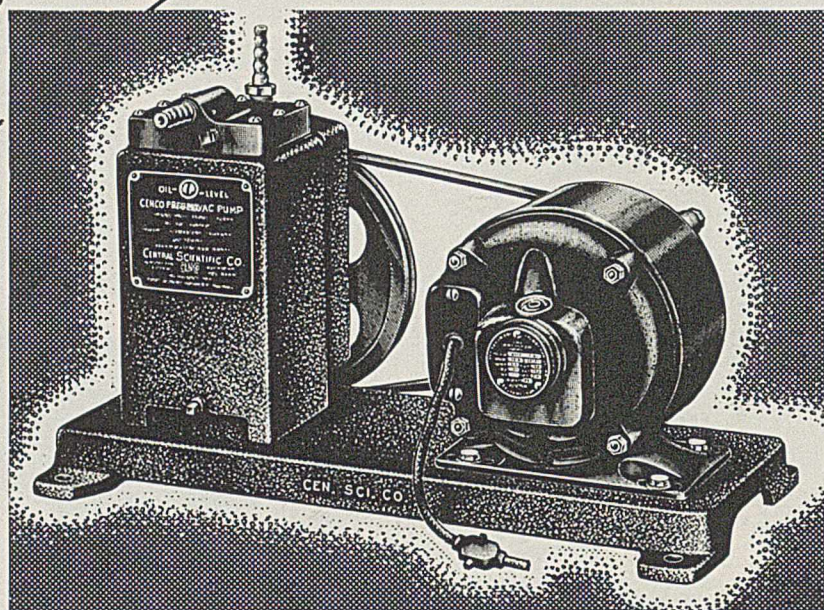
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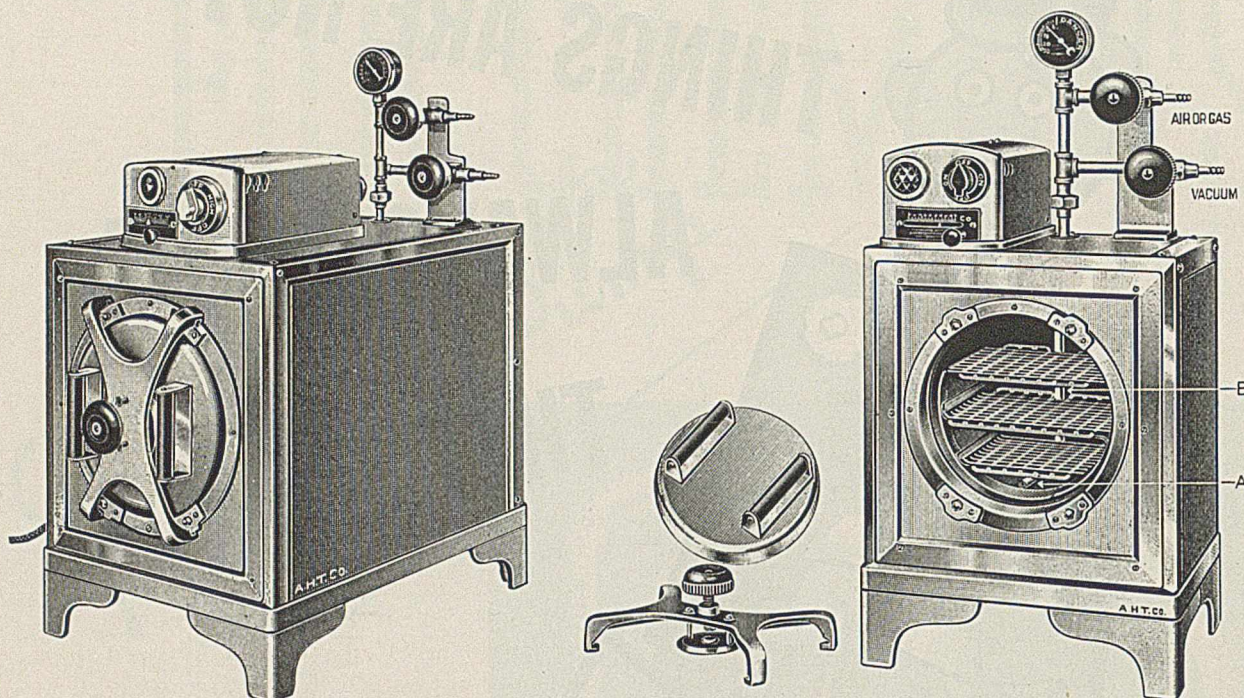
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| 7886. | Weber Electric Vacuum Oven, Small Model, as above described, outside dimensions 13 ¹ / ₂ × 12 ¹ / ₂ × 10 ¹ / ₂ inches, with vacuum chamber 8 inches deep × 7 ³ / ₄ inches diameter, with three shelves. Maximum current consumption 500 watts. With thermometer. For 110 volts a.c. | 200.00 | Code Word
Lydha |
| 7888. | Ditto, Large Model, outside dimensions 16 ¹ / ₂ × 15 ³ / ₈ × 12 ¹ / ₄ inches, with vacuum chamber 9 ¹ / ₂ inches deep × 9 ¹ / ₂ inches diameter. Maximum current consumption 800 watts. For 110 volts a.c. | 275.00 | Lyean |
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INDUSTRIAL and ENGINEERING CHEMISTRY

ANALYTICAL EDITION

Harrison E. Howe, Editor

Determination of High Viscosities

By Means of the Gardner Mobilometer

E. L. BALDESCHWIELER AND L. Z. WILCOX, Standard Oil Development Co., Linden, N. J.

A setup for Gardner mobilometers is described, whereby accurate temperature control and easy operation are obtained. The conclusions of Cornthwaite and Scofield that the correlation between absolute viscosity and mobility is a straight line have been checked by the authors for much higher vis-

cosities, at various temperatures, and for different disks. Provided rigid control of time and temperature is obtained and improvements in mechanical construction are made, the mobilometer can be used as a precision instrument for the determination of absolute viscosity.

THE mobilometer, originally described by Gardner and Parks (3), was designed to be used as a production control instrument to secure uniformity in consistency between different batches of a given product. The instrument was first recommended for control in paint and lacquer manufacturing. Later, Gardner and Van Hueckeroth (4) extended its use to the testing of food products, mineral oils, vaseline, and coal tar. The instrument has also been described at length by Sward and Stewart (6). While no claim for high accuracy of results obtained with the instrument was made by Gardner and Parks (3), Cornthwaite and Scofield (2) showed that under rigid control of temperature and time the apparatus gave a remarkably close correlation with the absolute viscosity of a number of samples possessing true fluid flow. This correlation resulted in a straight line passing through the origin when plotted on rectangular coordinates.

The work of Cornthwaite and Scofield (2) was, however, limited to oils of relatively low viscosities (about 8 poises). It was necessary for this laboratory to determine a number of mobilities and viscosities of much higher values with a fair degree of accuracy. For this purpose the relationship between viscosity and mobility for true fluids was obtained at various temperatures, using oils of much higher viscosities. The apparatus is described below.

Apparatus

In order to obtain flexibility and, at the same time, accurate temperature control, the barrel of the mobilometer was provided with a brazed outer brass jacket about 0.5 inch wide fitted with outlets at the top and bottom. These outlets are connected by means of rubber tubing to the circulation outlets of a Hoeppler (5) thermostat which is capable of controlling the temperature to within $\pm 0.02^\circ$ F. This apparatus provides a very flexible control of the temperature and, by using the proper circulation fluid and regulator in the Hoeppler thermostat, it is also suitable for low-temperature work, as shown by the authors in a previous article (1). The necessity of immersing the whole mobilometer in a bath is thereby avoided, which is an important advantage when working at extreme temperatures.

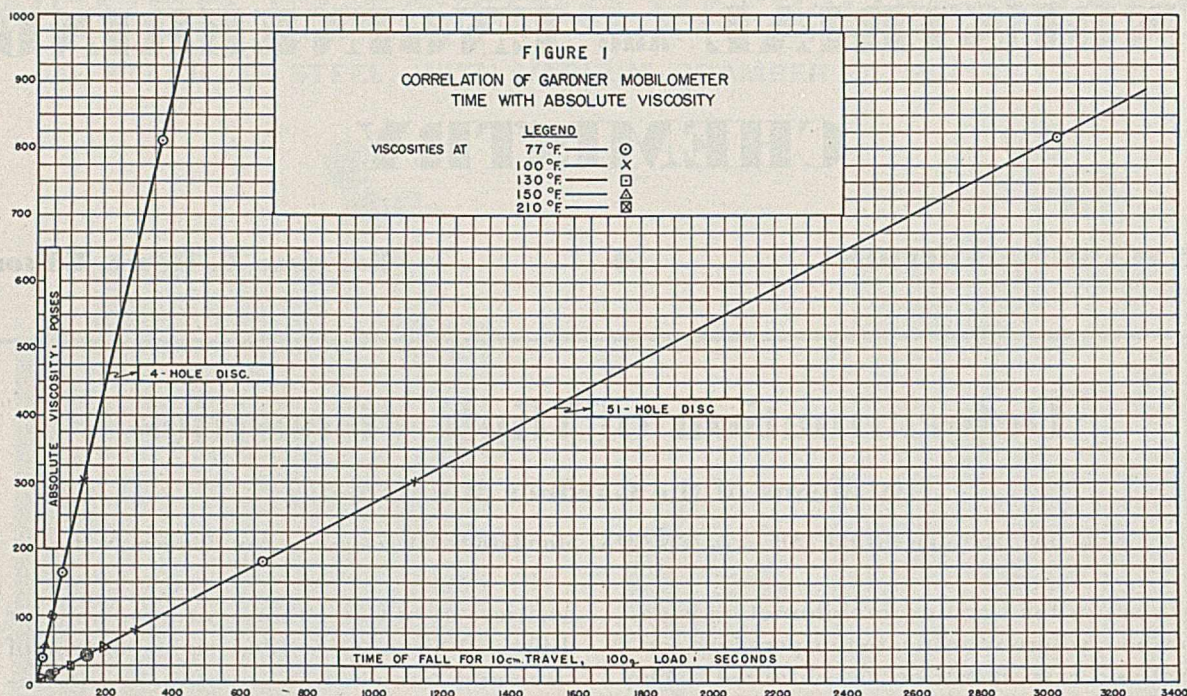
Timing in this laboratory is obtained by Veeder-Root magnetic counters, reading directly in tenth seconds. These are run by a contactor connected to an American Time Products constant-frequency generator. This generator, which is run by the plant power, allows a fluctuation of ± 10 volts and a frequency variation of ± 2 cycles in the current. Under these conditions the frequency does not vary by more than ± 0.001 cycle. This arrangement has given complete satisfaction, the frequency fluctuations having seldom exceeded the above limits. A more complete description of this equipment will be published in the near future. Viscosities were carried out by means of Ubbelohde (7) suspended level viscometers. A No. 4 capillary (constant $C = 10.04$) was generally used, except for the lower viscosities which were obtained with a No. 3 capillary (constant $C = 1.015$). The results thus obtained were in kinematic units. The absolute viscosities were calculated by multiplying the kinematic results by the density (obtained by pycnometer) of the individual samples at the various temperatures. The above procedure will give kinematic viscosities within ± 0.2 per cent while the densities were accurate to about 0.001. The constant-temperature bath used for the determination of kinematic viscosities can be controlled to within $\pm 0.02^\circ$ F.

Results

The choice of materials available for the determination of viscosities by means of viscometers of the glass capillary type is limited by the following considerations:

1. The material must have true viscous flow—i. e., the rate of shear should be proportional to the shearing stress.
2. It must be a true solution, absolutely free of suspended particles.
3. It must be transparent, so that a sharp meniscus can be seen when determining the viscosity.

The heaviest Pennsylvania bright stock available had a viscosity of about 163 poises at 77° F. In order to obtain high viscosities, especially at the higher temperatures, it was necessary to prepare blends of the above mineral oil with various amounts of an isobutylene polymer of very high molecular weight. Thus viscosities as high as 800 poises at 77° F. and 300 poises at 150° F. were obtained. Determina-



tions of both viscosities and mobilities were carried out at 77°, 100°, 130°, 150°, and 210° F. Mobilities were also carried out with both a 51-hole and a 4-hole disk. The results are shown in Figure 1, which is self-explanatory. (Concentric rings indicate determinations made on different materials having the same viscosities.)

Conclusions

From an examination of the curves shown in Figure 1, the following conclusions can be drawn:

The correlation between Gardner mobilometer time (in seconds) and absolute viscosity (in poises) is a straight line passing through the origin.

The above statement holds true for determinations made at various temperatures, and for different disks, although, as should be expected, changing the disk changes the slope of the curves. In other words, each disk has its own curve passing through the origin.

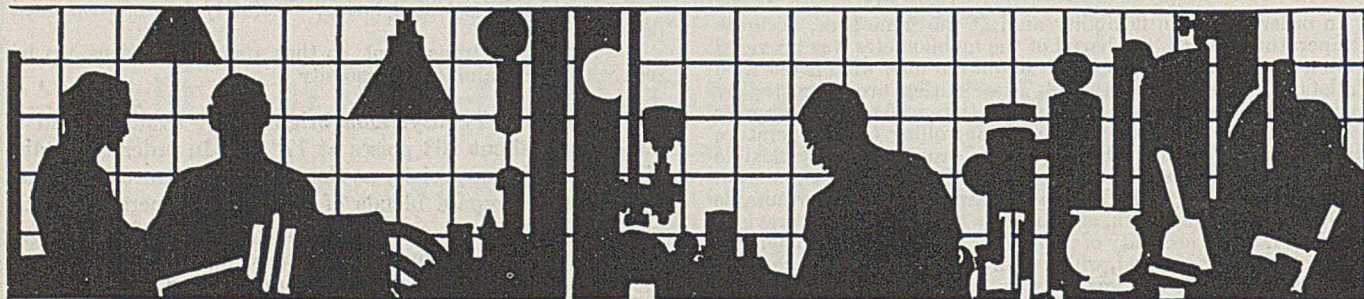
The results check the work and conclusions of Cornthwaite and Scofield (2) in every respect. However, the slope of the curve obtained by these authors for their 51-hole disk is not quite the same as that presented in this paper. This is due to the fact that it is mechanically impossible to manufacture disks identical in all respects; small variations in the shape of the disk as well as in the sizes and spacing of the holes will

cause changes in mobilities and thus alter the slope of the curve.

While admittedly the mobilometer is primarily a control instrument, it is susceptible of mechanical improvement. It is based on sound principles and the data presented show that it can be used for the determination of viscosity, being particularly useful for opaque materials of very high viscosities. It is only necessary to calibrate each disk against a material of known viscosity at any given temperature, plot this point, and draw a straight line through the origin. The sizes and number of holes in a given disk can be varied at will to suit any particular case.

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Phenols in Low-Temperature Tar

THOMAS B. SMITH AND LEO KASEHAGEN

Coal Research Laboratory, Carnegie Institute of Technology, Pittsburgh, Penna.

VARIOUS workers have identified individual dihydric phenols in the products of the low-temperature carbonization of bituminous coals, including resorcinol (13, 14), hydroquinone (13, 14), catechol (1, 2, 3, 5, 9, 11-17), and some of the homologs of catechol (4, 13, 14), but ratios of the dihydric to the monohydric phenols produced during carbonization have not been reported. This paper describes a method by which this ratio, or, more specifically, the average number of hydroxyl groups per molecule of the phenols of a tar, may be determined, and gives the results obtained by applying it to the tars produced by carbonizing several bituminous coals at temperatures from 400° to 600° C.

The usual method of analysis, which involves the double distillation of the tar and alkali extraction of the distillate in the presence of air, is not satisfactory for the determination of the quantity of dihydric phenols present in a tar, because of the instability of these phenols toward heat and oxidation, particularly in the presence of alkali. The double distillation causes sufficient decomposition of these phenols to preclude the possibility of an accurate quantitative determination of their presence in the tar as originally produced. The alkali extraction of the tar distillate in the presence of air continues the decomposition to such an extent that by the time the phenols have been separated from the other constituents of the tar, only a small portion of the dihydric phenols may be left. In the present method, the tar is not distilled. The quantity and type of the phenols as they exist in the whole tar, rather than in the tar distillate, are determined.

Briefly, the method consists of extracting a tar with alkali in an inert atmosphere to prevent oxidation, methylating the alkali phenolates to stabilize the hydroxyl groups, and recovering the methylated phenols. From the methoxyl percentage and average molecular weight of these latter, their average number of methoxyl groups per molecule is calculated. This value also represents the average number of hydroxyl groups per molecule of the phenols originally in the tar. From it the ratio of dihydric to monohydric phenols may be calculated, provided it be assumed that dihydric phenols are the only polyhydric ones present.

Carrying out the alkali extraction of the tar, and the subsequent methylation, in an atmosphere of nitrogen was effective in preventing oxidation of the tar. When a 500° tar from a Pittsburgh Seam coal was extracted with alkali in the presence of air, an appreciable quantity of a solid decomposition product was precipitated out on the sides of the separatory funnel. When a similar extraction was carried out in an atmosphere of nitrogen, this evidence of decomposition was absent. The alkali extraction offers another difficulty, however, in that the alkaline solution dissolves not only phenols but neutral material as well, because of the solubility of hydrocarbons in the solution of alkali phenolates. This difficulty was overcome by extracting the alkali phenolate solution with benzene, again carrying out the extraction in an atmosphere of nitrogen. The benzene, however, removed not only the hydrocarbons, but also a small quantity of the phenols. These phenols were recovered by extracting the benzene solution with fresh alkali. There was slight tendency for the hydrocarbons to redissolve in the alkali, as their solubility in the alkaline solution depends upon the presence of appreciable quantities of alkali phenolates.

The methylation was effected by means of dimethyl sulfate, which apparently gave a complete reaction when used in

excess. The excess was destroyed with alkali, which also hydrolyzed any esters that may have been formed from the carboxylic acids in the alkali phenolate solution. The methylated tar phenols are much more stable than the corresponding phenols. They are not so unstable toward heat as the phenols, and show little tendency toward oxidation when standing in air. This last property made it possible to discontinue the use of an inert atmosphere as soon as the methylation was completed, and permitted the methoxyl percentage and the average molecular weight of the methylated phenols to be determined with little danger of decomposition during the necessary handling.

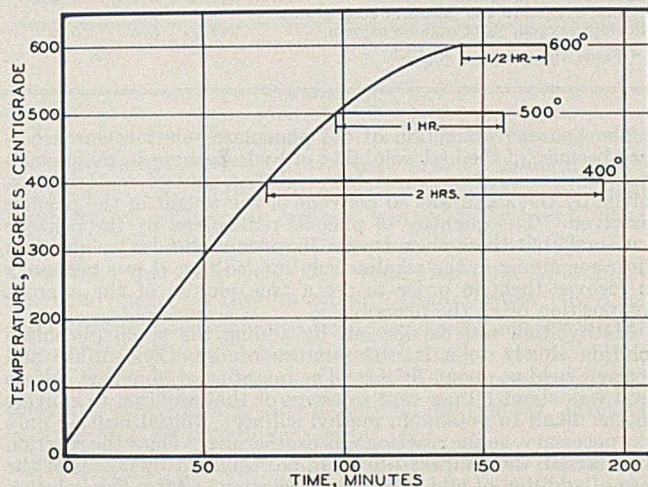


FIGURE 1. TEMPERATURE PROGRAMS FOR CARBONIZATIONS IN THE FISCHER ALUMINUM RETORT

The entire method was applied to a mixture made up of 2,6-xylolol, tetralin, *p*-cymene, *n*-heptane, cyclohexanol, biphenyl, *o*-toluidine, and acetic acid. The methylated xylolol was obtained in a pure form with a yield of better than 93 per cent of theory. The methylation step alone was checked by methylating catechol. The veratrole resulting amounted to 97 per cent of theory.

Experimental

The tars analyzed in this study were produced in a Fischer aluminum retort (10). Each charge consisted of 100 grams of the coal, ground to 20- to 60-mesh size. The retort was subjected to the temperature programs shown by the curves of Figure 1. For each carbonization the sample was heated rapidly to the desired temperature and maintained at this temperature until there was no visible evidence of distillation of tar from the exit tube of the retort. Throughout the heating, nitrogen was passed into the retort at a rate of 20 cc. per minute. The tar and liquor were collected in a small side-arm separatory funnel immersed in crushed ice. As soon as a carbonization was completed, the receiver was stoppered and weighed to determine the amount of tar plus liquor formed. A source of nitrogen was then connected to the side arm of the receiver, and the tar and liquor were extracted successively with 15-, 10-, and 10-cc. portions of 5.5 *N* potassium hydroxide. The solution of phenolates was extracted with three 20-cc. portions of benzene, which was then extracted with two 10-cc. portions of 5.5 *N* potassium hydroxide. All the extractions were carried out in an atmosphere of nitrogen. The alkaline solutions were combined, and enough 15 *N* potassium hydroxide was added to make the solution 6 *N*, allowing for the diluting effect of the liquor that was formed with the tar.

TABLE I. ANALYSES OF COALS USED

Seam	Mine	Bituminous Rank	Proximate Analysis ^a			Moisture in Sample Carbonized %	Ultimate Analysis ^b				
			Volatile matter %	Fixed carbon %	Ash %		C %	H %	N %	S %	O %
Pittsburgh	Edenborn	High-volatile A	33.6	57.0	7.5	1.4	85.0	5.7	1.7	0.7 ^c	6.9
Illinois No. 6	Orient No. 1	High-volatile B	33.9	50.3	9.1	8.2	80.6	6.1	2.1	0.7 ^c	10.5
High Splint	Clo-Splint	High-volatile A	36.4	57.3	3.4	3.9	83.8	5.7	1.8	0.6 ^d	8.1

^a Analyses of mine and tippel samples, courtesy of U. S. Bureau of Mines, Pittsburgh Experiment Station.

^b Dry, mineral-matter-free basis (mineral matter = 1.1 times ash).

^c Organic sulfur only.

^d Total sulfur.

TABLE II. ANALYSES OF TARS PRODUCED IN FISCHER RETORT

Carbonizing temperature, ° C.	High Splint Coal			Edenborn Coal			Illinois No. 6 Coal						
	400	500		400	500		600	400		500		600	
Tar yield, grams ^a	8.3	16.5	16.2	5.0	14.2	13.8	14.5	6.2	6.4	13.4	13.5	12.9	12.9
Liquor yield, grams ^a	5.6	6.2	6.1	2.3	3.9	3.8	3.7	11.0	11.4	11.2	11.2	13.2	13.2
Methylated phenols, yield, grams ^a	2.0	4.7	4.7	0.6	3.7	3.4	3.6	2.6	2.9	5.0	5.1	5.4	5.4
Methylated phenols, % MeO	17.50	17.86	17.96	18.53	16.89	16.86	16.72	22.23	21.84	19.10	18.98	20.76	20.76
Methylated phenols, molecular weight	192	181	181	207	206	207	202	173	174	165	168	154	154
Average hydroxyls per phenol molecule	1.084	1.043	1.049	1.237	1.123	1.127	1.091	1.241	1.227	1.017	1.029	1.031	1.031
Dihydroxy compounds in phenols, %	8.4	4.3	4.9	23.7	12.3	12.7	9.1	24.1	22.7	1.7	2.9	3.1	3.1
Phenols, yield, grams ^a	1.8	4.3	4.3	0.55	3.4	3.1	3.3	2.3	2.6	4.6	4.7	4.9	4.9
Phenols, yield, % of tar	21.7	26.0	26.5	11.0	24.0	22.4	22.8	37.1	40.6	34.3	34.8	38.0	38.0
Phenolic oxygen, grams ^a	0.182	0.433	0.435	0.057	0.322	0.296	0.311	0.298	0.326	0.494	0.500	0.579	0.579
Organic oxygen in 100 grams of coal, grams		7.50			6.34					8.68			
Phenolic oxygen, % of organic oxygen	2.4	5.8	5.8	0.9	5.1	4.7	4.9	3.4	3.8	5.7	5.8	6.7	6.7

^a From 100 grams of coal.

The benzene extraction of the phenolate solution was necessary because of the high solubility of hydrocarbons in the phenolate solution. In one case the weight of neutral material dissolved by the alkali was 40 per cent of the weight of the phenols dissolved. The quantity of phenols redissolved by the benzene was small, but these phenols were lower in molecular weight than those remaining in the alkaline solution, so that it was necessary to recover them in order to get a true picture of the average composition of all the phenols.

Methylation was carried out by adding the alkali phenolate solution slowly to a stirred mixture of dimethyl sulfate and benzene held at about 70° C. The quantity of dimethyl sulfate used was about 50 per cent in excess of that required to convert all the alkali to potassium methyl sulfate. Initial heating only was necessary, as the reaction was exothermic. Once the reaction was started, the temperature could be regulated by means of the rate of addition of alkali phenolate solution. After this solution had been completely added, twice the amount of 15 N potassium hydroxide equivalent to the remaining dimethyl sulfate was added to the hot methylating mixture. After the destruction of the excess dimethyl sulfate, two layers remained. The aqueous layer contained the salts of the carboxylic acids originally in the tar, and the benzene layer the methylated phenols. This layer was washed several times with alkali and water, and dried, and the benzene was removed by distillation followed by evacuation.

The methoxyl percentage of the methylated phenols was determined by a modified Vieböck micromethod. Analyses for sulfur were made to ascertain whether the dimethyl sulfate, which would raise the methoxyl percentage, had been completely removed from the methylated phenols. Their average molecular weight was determined cryoscopically using biphenyl as a solvent. Apparent molecular weights were determined for several different concentrations, and the value obtained by extrapolating to zero concentration taken as the true average molecular weight. The molecular weights of several pure aromatic ethers were determined by this method with errors of less than 2 per cent, and checks within 4 per cent were obtained on the methylated phenols from the tars.

Analyses of the coals used are shown in Table I. The Pittsburgh Seam coal is a high-volatile A coking coal, the Illinois No. 6 coal is high-volatile B and has a high oxygen content, which barely falls within the limits for coking coals. The High Splint coal is likewise high-volatile, but in contrast to the other two is dull and contains a high percentage of attritus.

Table II gives the results of the analyses of the tars produced under the indicated conditions from the various coals. Yields of tar and liquor were determined in separate carbonizations, as no separation of tar from liquor was made during the analyses. The average numbers of hydroxyl groups per phenol molecule were calculated from the methoxyl percentages and average molecular weights of the methylated

phenols, shown just above in Table II. The percentages of the phenols which are dihydric, assuming that there are no trihydric or higher phenols present, follow directly. This assumption is based on the facts that the probability of forming trihydric phenols is much lower than that of forming dihydric phenols, and that the presence of trihydric phenols in tar has never been reported. The yields of phenols, as calculated from the experimentally determined yields of methylated phenols, and the phenolic oxygen, as per cent of the organic oxygen in the coal, are also shown in Table II.

Discussion of Results

The high ratios of dihydric to monohydric phenols in the tars made from Edenborn coal are the most striking feature of the results. The ratio decreased as the temperature of carbonization was raised (see Figure 2). This change may be accounted for if the known instability toward heat of the dihydric phenols is considered. The tendency to produce oxygenated compounds high in polyhydric phenols appears to be characteristic of this Pittsburgh Seam coal. This tendency, however, cannot be linked to the classification of the coal, on the basis of the present data. The High Splint coal, which differs from Edenborn coal mainly in that it is a dull instead of a bright coal, gave less than half the yield of dihydric phenols that was obtained from Edenborn coal.

The Illinois No. 6 coal, because it has a higher oxygen content, might be expected to produce phenols having a larger average number of hydroxyl groups per molecule than those from Edenborn coal. The increased oxygen content might increase the probability of two oxygen atoms being attached to the same nuclear grouping. However, when the size of these nuclear groupings as possibly indicated by the molecular weights of the phenols produced from the two coals is considered, these probabilities for the two coals are not so very different. The nuclear groupings in Edenborn coal must definitely be larger than those in the Illinois coal because the molecular weights of the phenols from Edenborn coal, as calculated from the experimentally determined molecular weights of the methylated phenols, are close to 190, while those from the Illinois coal are only 150. This difference in nuclear size almost compensates for the effect of the difference in oxygen contents when considering the probability of having two oxygen atoms linked to the same nucleus. The higher oxygen content of the Illinois coal is, however, reflected by

the increased productions of phenols, and by the fact that the phenolic oxygen from the Illinois coal represents a larger percentage of the organic oxygen of the coal than is the case with the phenols from Edenborn coal.

The very sharp drop from 23.4 to 2.3 in the percentage of dihydric phenols in the total phenols, as the temperature of carbonization was raised from 400° to 500° C., for the Illinois coal may perhaps be explained on the basis of the instability toward heat of the dihydric phenols. However, this drop in the same temperature interval for the dihydric phenols from Edenborn coal was only from 23.7 to 12.5 per cent. Thus it may be assumed that the dihydric phenols produced from the two coals are different. These phenols are not uniform in their stability toward heat. For example, of the three dihydroxybenzenes, the meta compound is decomposed more rapidly than either of the other isomers, when pyrolyzed at 350° C. (18). If the oxygen distribution in Illinois coal is such that only very thermally unstable dihydric phenols can be produced, then only a very low temperature of carbonization will permit this production to take place.

In Table III is given a comparison between the yields of phenols obtained by the Bureau of Mines (7, 8) using a standard method of analysis (6), and the yields obtained by the method described in this paper. The latter are uniformly higher than the ones given by the standard method, and indicate that appreciable amounts of the phenols originally present in the tar are either destroyed during the standard analysis, or else remain in the pitch, since in the standard method only the phenols in the tar distillate are determined.

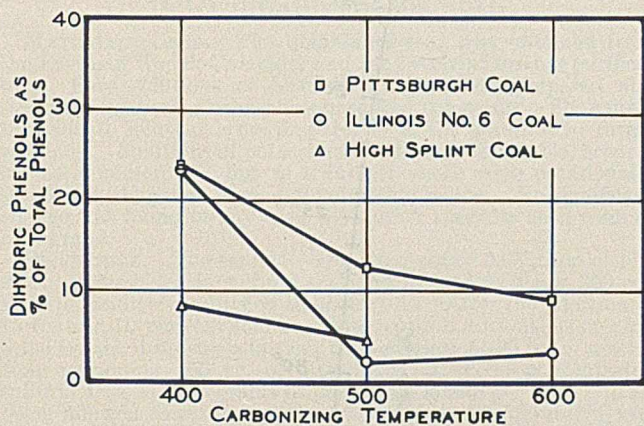


FIGURE 2. COMPOSITION OF PHENOLS AS A FUNCTION OF CARBONIZING TEMPERATURE

This conclusion is open to the objection that the tars made by the Bureau of Mines, and those studied in this investigation, were produced in different types of retorts and therefore are not necessarily similar. For this reason, the phenols in a 500° tar from Edenborn coal were determined by the three methods indicated in Table IV with the results there given. It is clearly shown that the method described in this paper (method 1) does give higher yields of phenols than the standard method (method 3). The results given by method 2 show that most of the decomposition of phenols in the standard method of analysis occurred during the double distillation. Not only was the quantity of phenols in the distillate smaller than that in the crude tar, but the nature of the phenols was also quite different. For example, in the crude tar the phenols had an average molecular weight of 213 and an average of 1.04 hydroxyls per molecule, while in the distillate obtained by the double distillation of the crude tar, they had an average molecular weight of only 149 and an average of 1.02 hydroxyls per molecule.

TABLE III. COMPARISON WITH RESULTS OF STANDARD METHOD OF ANALYSIS

Temperature of carbonization, ° C.	Pittsburgh Coal		Illinois No. 6 Coal	
	500	600	500	600
Tar acids by standard method, % of dry tar	17.4	15.8	29.8	30.0
Phenols by method described in this paper, % of dry tar	23.2	22.8	34.5	38.0

TABLE IV. PHENOLS IN A 500° TAR FROM EDENBORN COAL

Method	Yield of Phenols, Per Cent of Dry Tar	Average Molecular Weight of Phenols	Average Number of Hydroxyls per Molecule
Method 1 ^a	20.7	213	1.04
Method 2 ^b	15.1	149	1.02
Method 3 ^c	14.2

^a Alkali extraction of crude tar in absence of air, followed by methylation of phenolate solution.

^b Double distillation of crude tar, with distillate treated as in method 1.

^c Double distillation of crude tar, with tar acids in distillate determined by contraction on shaking with alkali.

Summary

A method for determining the quantity and average number of hydroxyl groups per molecule of the phenols of a low-temperature tar has been developed and applied to tars made from several bituminous coals at temperatures from 400° to 600° C. The results on a tar from a Pittsburgh Seam coal show that this coal yields tar containing large amounts of dihydric phenols. Results on tars from Illinois No. 6 coal show that this coal can also yield tars containing large quantities of dihydric phenols if the carbonization temperature is kept sufficiently low, but that as the temperature is increased these dihydric phenols are not found in the tar, probably because of their instability toward heat.

Yields of phenols obtained by this method are higher than those obtained by the standard method of analysis, because of decomposition during the double distillation, and probably also because of failure of the phenols in the crude tar to distill at the temperatures reached in the distillation.

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Barometric Correction Nomograph for Hydrogen Electrode

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MEASUREMENT of pH with certain types of hydrogen electrodes must include a correction for pressure of the hydrogen when it differs from standard one atmosphere of 760 mm. of mercury. The hydrogen pressure may not be standard because of barometer fluctuation, the vapor pressure of solution, hydrostatic head, or other reasons. The correction for these, which Clark (1) calls " $E_{\text{barometer}}$ ", may easily be evaluated, and tables for selected pressures and temperatures are included by Clark (1), Kolthoff (2), and others.

A nomograph, once constructed, offers a convenient method of evaluating this barometric correction for any pressure and temperature within its range; it avoids interpolation in the tables and gives a precision satisfactory for most purposes. Such a nomograph is given here, and as a further convenience, has been made to include the correction for thermal expansion of the mercury in the barometer.

This chart is limited to use when the barometer has a

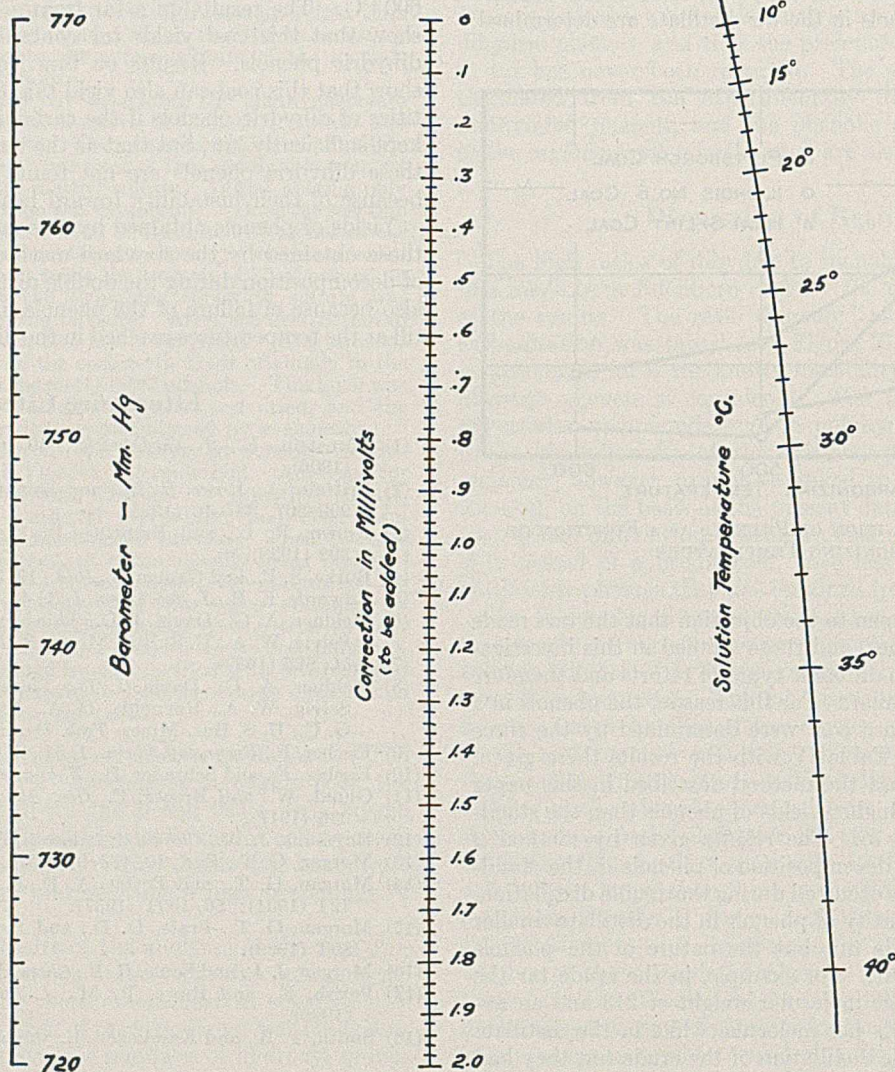
brass scale and is at approximately the temperature of the solution. When the two temperatures are not identical, a small error may be introduced, but the difference must be nearly 5° C. before an error of 0.01 millivolt results. Hydrostatic head and vapor pressure lowering by the solute, neglected here, are usually more important than this.

Acknowledgment

Data for construction of this nomograph were taken from Clark (1). Thanks are due W. W. Smith and Daniel Rodier for assistance in calculation and drawing.

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NOMOGRAPH

Determination of Bismuth by the Quinaldine Salt of Iodobismuthous Acid

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IT HAS long been known that many organic bases, such as quinoline (4), quinine (3) and the like, form, in the presence of iodides and certain metals, compounds which because of their characteristic colors are suitable for the detection of these metals. However, very few of these organic bases have been investigated from a quantitative standpoint. Berg and Wurm (2) were able to determine cadmium and bismuth, as well as separate these metals from others, by the use of naphthoquinoline and *o*-hydroxyquinoline. The latter reagent was also studied by Kolthoff and Griffith (6). In view of the many compounds available, some of which are more basic than *o*-hydroxyquinoline, it seemed advisable to investigate this field further.

Quinaldine was found to give good results in the determination of bismuth. In the presence of dilute sulfuric acid and excess of potassium iodide, the bismuth is completely precipitated by this reagent, and may then be readily determined volumetrically, through the relationship of four iodides to one bismuth. Many other ions, both metallic and nonmetallic, either do not affect the determination at all or may be readily prevented from interfering.

Determination of Bismuth

MATERIALS USED. The quinaldine used was obtained from the Eastman Kodak Company and used without further purification. Pure solutions of bismuth nitrate were prepared and standardized both as the phosphate and the oxide. The potassium iodate solutions were prepared from the pure salt by direct weighing. Solutions of potassium iodate are very stable, according to Jamieson (5), but as a further check were occasionally standardized against pure, dry potassium iodide. No significant change was found in any case. All other reagents used were of C. P. grade.

PROCEDURE. The sample (containing about 0.03 gram of bismuth) is dissolved in sulfuric acid and diluted to about 200 cc., and the acidity is adjusted to about 1 *N*. After the addition of 15 cc. of a 10 per cent sodium sulfite solution, bismuth is precipitated by the dropwise addition, with stirring, of 20 cc. of a solution containing 150 cc. of quinaldine, 50 cc. of concentrated sulfuric acid, and 75 grams of potassium iodide per liter. After being allowed to settle, which requires 15 to 20 minutes if well stirred, the red precipitate is filtered with suction on a Gooch filter or a fritted-glass filter cell. The precipitate may stand several hours before filtering without apparent decomposition, but preferably not overnight. It is first washed with 40 to 50 cc. of a solution of 35 cc. of quinaldine, 15 cc. of concentrated sulfuric acid, and about 0.8 gram of potassium iodide per liter. As washing with water must be avoided to prevent decomposition of the precipitate, the small amount of excess iodide left by the first wash solution is removed by washing with 30 cc. of a solution of 10 per cent acetone in *N* dibutyl ether.

The crucible containing the well-washed precipitate is then transferred to a beaker and about 100 cc. of a 5 per cent sodium hydroxide solution are added. To ensure complete decomposition of the precipitate the solution is heated almost to boiling for about 20 minutes, after which it is cooled and neutralized with concentrated hydrochloric acid, and an excess of 10 cc. is added. After adding 8 cc. of a 0.5 *M* potassium cyanide solution, the iodide is titrated to iodine cyanide according to Lang's method (7). Each milliliter of the 0.1 *N* (0.025 *M*) potassium iodate solution is equivalent to 0.002612 gram of bismuth.

An average error of about 0.30 per cent was found in analyzing pure bismuth samples by this method.

Separation of Bismuth from Mixtures

With slight modifications the procedure described above may be used for the determination of bismuth in the presence

of a considerable number of other metallic ions and many of the common anions.

ANTIMONY. In the case of antimony a synthetic sample was prepared containing 0.044 gram of antimony sulfate and 0.0303 gram of bismuth. Before precipitating the bismuth about 4 grams of ammonium tartrate were added and the bismuth was determined as described above. The average value for six such determinations was 0.0304 gram of bismuth, with an average error of 0.29 per cent.

LEAD. To the standard bismuth solution, containing 0.0303 gram of bismuth, was added 0.08 gram of lead nitrate. The bismuth may be precipitated directly, after the usual dilution and acidification with sulfuric acid, but it was found to be slightly more accurate to remove the lead first. This was done by adding excess sodium sulfate and filtering off the precipitated lead sulfate. The average of six such determinations was 0.0303 gram of bismuth, with an average error of 0.27 per cent.

CADMIUM. A composite sample, containing 0.0303 gram of bismuth and 0.08 gram of cadmium chloride, was analyzed for bismuth after the addition of 5 cc. of pyridine. The average of six analyses was 0.0303 gram of bismuth, with an average error of 0.29 per cent.

COPPER. In this case a sample was prepared to consist of 0.0303 gram of bismuth and 0.075 gram of cupric sulfate. Before precipitating the bismuth, 15 cc. of a 10 per cent sodium sulfite solution were added. The average of eleven such determinations was 0.0303 gram of bismuth, with an average error of 0.14 per cent.

IRON. The separation of 0.0303 gram of bismuth from 0.22 gram of ferric nitrate offered no difficulty. The bismuth was precipitated in the presence of about 3 grams of sodium sulfite. Seven such determinations gave an average value of 0.0303 gram of bismuth, with an average error of 0.18 per cent.

TIN. The determination of bismuth in the presence of stannous tin gave no trouble. In this case the average of eight determinations of 0.0303 gram of bismuth in the presence of 0.80 gram of stannous chloride was 0.0303 gram of bismuth. If the tin was present in the stannic form, it was found best to add about 15 cc. of 10 per cent sodium sulfite solution prior to precipitation of the bismuth. The average of six determinations of 0.0303 gram of bismuth in the presence of 0.90 gram of stannic chloride was 0.0305 gram of bismuth, with an average error of 0.43 per cent.

ARSENITE. In this case 0.0303 gram of bismuth was determined in the presence of 0.04 gram of arsenic trioxide, proceeding exactly as for a pure bismuth sample. The average of six determinations was 0.0304 gram of bismuth, with an average error of 0.30 per cent.

ARSENATE. The determination of 0.0303 gram of bismuth in the presence of 0.036 gram of arsenic acid presented no difficulty. Proceeding as for a pure bismuth sample, the average of six analyses was 0.0303 gram of bismuth, with an average error of 0.05 per cent.

PHOSPHATE. The procedure in the presence of phosphate was the same as for a pure bismuth sample, with one exception. Here the acidity was adjusted to about 4 *N*, in order to prevent the precipitation of any bismuth as the phosphate. Under these conditions the average of six determinations of 0.0303 gram of bismuth in the presence of 0.20 gram of sodium orthophosphate was 0.0303 gram of bismuth, with an average error of 0.10 per cent.

NICKEL, CHROMIUM, COBALT, MANGANESE, CALCIUM, BERYLLIUM, URANYL, ALUMINUM, TITANIUM, AND BARIUM. A synthetic sample was prepared containing 0.0303 gram of bismuth and 0.10 gram of chromium nitrate, 0.06 gram of manganese sulfate, 0.05 gram of cobalt sulfate, 0.08 gram of calcium nitrate, 0.16 gram of beryllium carbonate, 0.03 gram of uranyl acetate, 0.05 gram of nickel sulfate, 0.125 gram of aluminum sulfate, 0.08 gram of titanium sulfate, and 0.04 gram of barium nitrate. The bismuth was then precipitated and determined exactly as in a pure sample. The average of seven such determinations was 0.0303 gram of bismuth, with an average error of 0.05 per cent.

ANTIMONY, ZINC, ALUMINUM, NICKEL, SODIUM, AND POTASSIUM. A composite sample was prepared to contain 0.0303 gram

of bismuth and 0.02 gram each of antimony, zinc, aluminum, nickel, sodium, and potassium, each present as the sulfate. Before precipitation of the bismuth, 4 grams of ammonium tartrate were added and the determination was completed as usual. The average of five such analyses was 0.0306 gram of bismuth, with an average error of 0.60 per cent.

LEAD, ANTIMONY, TIN, AND COPPER. A synthetic lead-base bearing metal was prepared by adding 0.0303 gram of bismuth to 0.8 gram of lead, 0.1 gram of tin, 0.1 gram of antimony, and 0.02 gram of copper. The sample was dissolved in sulfuric acid, the lead sulfate was filtered off, 4 grams of ammonium tartrate and 10 cc. of 10 per cent sodium sulfite solution were added, and the bismuth was precipitated and determined as usual. The average of three such determinations was 0.0302 gram of bismuth, and the average error was 0.22 per cent.

Determination of Very Small Amounts of Bismuth

For the determination of very small amounts of bismuth the procedure previously outlined is slightly modified.

The precipitation of the bismuth is done in the same manner, except that smaller amounts of quinaldine and potassium iodide are used. The precipitate is filtered, washed, and dissolved as usual, but the titration in this case is done with 0.01 *N* (0.0025 *M*) potassium iodate solution (1 cc. = 0.0002612 gram of bismuth). The iodide is titrated to iodine chloride according to Andrews' method (1), which, in accordance with the findings of Kolthoff and Griffith (6) gives better results. A microburet was used in these titrations. The average of twelve samples, each containing 0.00032 gram of bismuth, was 0.00033 gram, with an average error of 2.3 per cent.

Interfering Ions

In the presence of any appreciable amount of chloride the results were generally low, in all probability because of a partial replacement of the iodide in the precipitate by chloride.

The largest amount of chloride present in any of the determinations reported was represented by 0.90 gram of stannic chloride.

Bismuth could not be determined in the presence of silver and mercury by this method.

Summary

A new method for the determination of bismuth, which involves the precipitation of bismuth as the quinaldine salt of iodobismuthous acid, is described. This method is applicable to amounts of bismuth as low as 0.3 mg.

By this method bismuth may be separated and determined in the presence of lead, antimony, tin, cadmium, copper, iron, chromium, manganese, cobalt, calcium, barium, uranyl, nickel, beryllium, aluminum, titanium, zinc, sodium, potassium, arsenite, arsenate, and phosphate.

The determination may be successfully carried out in acidities ranging from 2.5 to 10 per cent of sulfuric acid by volume.

The determination of bismuth in the presence of mercury and silver is not feasible. High concentrations of chlorides lead to low results.

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Direct Determination of Alumina in Certain Silicates

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THE direct determination of alumina in silicates, without regard to the other constituents, is often of considerable interest to the analytical chemist. Under ordinary conditions the determination follows a preliminary separation of silica by the conventional alkali carbonate—mineral acid solution—double dehydration procedure. If silica is not desired and volumetric means of evaluating the alumina content are to be used, single dehydrations suffice, provided that the acid-insoluble oxychlorides of the R_2O_3 group are added to the main silica filtrate. This procedure is extremely lengthy.

Knowles and Redmond (10) used a Berzelius decomposition for the removal of silica in feldspar, ultimately determining aluminum volumetrically as quinolate. Certain precautions are necessary to ensure complete removal of hydrofluoric acid, for the presence of this acid, even in minor quantities, prevents the complete precipitation of the aluminum complex. Excellent results are to be obtained by the use of this procedure.

Stuchert and Meier (18), who claim that the Berzelius decomposition leads to low results, modified the method to some extent. The sample is evaporated with hydrofluoric acid alone, and the residue is heated gently to remove the free acid and finally taken into solution by fusion with potassium pyrosulfate. The fused mass is dissolved in 1 to 10 sulfuric acid and the aluminum is determined as quinolate. Experience with the method in the author's laboratory indicated a partial loss of aluminum, and

there was some evidence of incomplete decomposition of the more refractory materials.

Procedures dependent upon removal of silica as silicon tetrafluoride find application only when the material under analysis is completely soluble in hydrofluoric acid or hydrofluoric-sulfuric acid. Ideal conditions for the determination of aluminum in silicates would be based upon (1) complete and rapid decomposition of the sample, (2) a volumetric determination, and (3) the presence of silica.

Inasmuch as silica does not form a complex with 8-hydroxyquinoline in the absence of a molybdic acid salt, it was thought that the aluminum could be precipitated directly from the acid solution of the alkali carbonate melt, as was first suggested by Krinke (12) for the determination of aluminum in glasses. Unfortunately, upon adjustment of the pH to the point of precipitation of aluminum as quinolate, the separation of silica presented a particularly difficult filtration problem and this mode of attack was abandoned. This separation of silica appears to be a function of the amount of aluminum present in the sample under analysis.

The rapid reaction with molten alkali and the amphoteric nature of aluminum oxide suggested an initial decomposition based upon fusion with potassium or sodium hydroxide, fol-

lowed by acidification to redissolve the aluminum hydroxide and precipitation of aluminum as the quinolate. Most silicates proved amenable to this fusion treatment, but separation of the silica, upon acidification of the water solution of the alkali melt, again caused difficulty.

Taking advantage of the property that 8-hydroxyquinoline removes the complex-forming metals from their insoluble salts (9), a method based upon the alkali fusion discussed above was devised. This involved fusion of the sample with an alkali flux in a nickel beaker, solution of the fused mass in water, and subsequent boiling to precipitate nickelic and ferric hydroxides. These were removed by filtration, and the clear solution was treated with an excess of an acetic acid solution of the reagent, the aluminum complex being soluble in the highly alkaline original solution. Hydrochloric acid was added, through the stage of complete precipitation, continuing until practically all of the precipitate was dissolved. The pH was finally adjusted with ammonium acetate. For some reason the presence of the organic reagent allows a greater acidity, before precipitation of silica, than does the modification previously outlined. This procedure was productive of excellent results when working with materials having a relatively low iron content—0.1 per cent or less.

Application of the method to the analyses of materials of relatively high iron content soon made it apparent that a portion of both the nickel and iron had escaped precipitation as hydrous oxide and was to be found in the filtrate. Quantitative determinations of the iron indicated that the amount escaping separation, probably as an alkali ferrate, was by no means constant. Previous work in the author's laboratory did not substantiate the broad statement of Stuchert and Meier (18) that iron to the extent of 3 per cent of the aluminum content is without effect upon the final results for aluminum. (Unpublished results of H. R. Shell show that ferric iron, introduced as ferric chloride into feldspar samples, precipitated quantitatively as the quinolate with the exception of a constant amount of 0.09 mg. which escaped as colloid. This value proved to be true for ferric oxide additions of 0.1 to 5.0 mg.)

The problem of iron interference presents itself most forcibly in the analysis of clays, unprocessed nepheline syenites, certain lepidolites, and other silicates most often bearing high-iron accessory minerals. Corrections based upon the ferric iron content of the alkali filtrate are not permissible, for the analyst has no assurance that the iron is to be found completely in the ferric condition at the time of precipitation of aluminum with 8-hydroxyquinoline. The analyst has two alternatives for the elimination of this difficulty: complete removal of iron before attempting precipitation of aluminum or the formation of a soluble iron complex which will inhibit the iron-quinolate reaction.

Both Haslam (6) and Skinner (17) remove iron as the sulfide from alkali tartrate solutions and Skinner has used this method as a basis for the determination of iron in glass sands. Fainberg and Tel (4) reduce the iron with sodium thiosulfate and form the stable alkali ferrocyanide by the addition of potassium cyanide. Heczko (7) uses a similar method, substituting hydrogen sulfide as the reductant. By either of the latter two methods aluminum can be determined in the presence of both silica and iron. Each, however, has the disadvantage inherent in the use of potassium cyanide in slightly acid solution.

Separations based upon controlled acidity (15, 20) or upon the formation of aluminum complexes with salicylic (1), malonic (2), or oxalic acid (21) all depend upon precipitation of iron as quinolate in acid solution and in some of the latter cases the carboxylic acid must be completely removed before the aluminum quinolate can be precipitated. These methods find their greatest usefulness in the separation of iron when

in relatively greater proportion than the aluminum. None would find application in the present instance because of the possibility of separation of silica at the optimum pH for the iron separation.

Churchill and Bridges (3) state that iron must be in the ferric condition before precipitation as quinolate can be accomplished and they resort to an oxidation before precipitation of iron and aluminum preliminary to the determination of beryllium in aluminum-beryllium alloys. It appeared that on reducing the iron to the ferrous state, precipitation of iron as quinolate would be prevented and reliable values for alumina would result. The ferrous salts proved unstable, however, as was shown by Willard and Tang (19), and precipitation of ferric quinolate invariably took place upon exposure of the previously reduced solutions to the atmosphere.

Ferrari (5) has recently shown that the ferrous complex of α, α' -bipyridine, $\text{Fe}(\text{C}_{10}\text{H}_8\text{N}_2)_3\text{X}_2$, where X is a monovalent acid radical, is so stable that the iron cannot be removed from this complex as the hydroxide, ferricyanide, or sulfide. Hill (8) has shown that, though the complex is easily dissociated in mineral acid solutions of high concentration, it is stable in the pH range of 3.5 to 8.5. Saywell and Cunningham (16) used the analogous complex-forming properties of *o*-phenanthroline for the same purpose, while Mayr and Gebauer (13) proposed the use of thioglycolic acid in ammoniacal solution to prevent the precipitation of iron as hydroxide. In all these the iron is previously reduced with sulfurous acid or an organic reductant such as hydroxylamine hydrochloride. These data indicated that an inhibition of the ferric quinolate precipitation might be possible if the iron were held as the ferrous complex. Early experimental trials proved this premise to be true with either *o*-phenanthroline or α, α' -bipyridine. The use of thioglycolic acid was not studied in this investigation. *o*-Phenanthroline may have some advantage, in the direction of greater stability (14), over the α, α' -bipyridine reagent.

Reagents and Equipment

c. p. pellets of sodium hydroxide are satisfactory.

Dissolve 100 grams of 8-hydroxyquinoline (Eastman Kodak Co.) in 200 ml. of glacial acetic acid and add to 3 liters of water previously heated to 80° C. Filter if necessary, and dilute to 4 liters.

Ammonium Acetate Buffer. Three grams of the salt per 10 ml. of solution.

One gram of *o*-phenanthroline or α, α' -bipyridine hydrochloride in 100 ml. of 6 N hydrochloric acid.

A saturated solution of hydroxylamine hydrochloride in water.

Potassium Bromate-Bromide, 0.25 N. Dissolve 6.97 grams of potassium bromate and 25 grams of potassium bromide in 1000 ml. of water. Standardize against sodium thiosulfate or by the method described by the author (11).

Sodium Thiosulfate, 0.10 N. Dissolve 25 grams of sodium thiosulfate pentahydrate and 1 gram of boric acid in 1000 ml. of sterile water. Store in a dark bottle. Standardize against iodine or by the method previously described (11).

Potassium Iodide, 60 per cent. Dissolve 60 grams of U. S. P. potassium iodide in 100 ml. of water.

Add 5 grams of a cold suspension of soluble starch to 500 ml. of boiling water. When cool add 15 grams of potassium iodide and 5 grams of sodium hydroxide in 25 ml. of water. Store in a dark bottle.

A 250-ml. nickel beaker of the same shape as the Griffin low-form glass beaker is suitable.

Procedure

A quantity of the dried (105° C.) and finely ground (200-mesh) sample sufficient to produce between 10 and 30 mg. of alumina is transferred to a nickel beaker containing at least ten times the sample weight of previously fused sodium hydroxide. The beaker is heated, gently at first, to complete solution of the sample in the flux and finally at 400° to 500° C. for a few moments; the beaker should show a dull red through the melt. The beaker is covered, and cooled, and 100 ml. of water are added. Heating to solution of the fused mass is followed by

TABLE I. DETERMINATION OF ALUMINA

Sample	Material	Al ₂ O ₃	Al ₂ O ₃	Differ- ence	Remarks
		Present Mg. ^a	Found Mg.		
1	Feldspar No. 70	18.03 ^b	18.03	±0.00	
2	Feldspar No. 99	19.06 ^b	19.06	±0.00	
3	Feldspar	15.60	15.60	±0.00	
4	Feldspar	18.20	18.20	±0.00	
5	Cornwall stone ^c	15.15	15.17	+0.02	1.2% F present
6	Lepidolite	13.40	13.40	±0.00	3.4% F present
7	Lepidolite ^c	13.64	13.64	±0.00	2.2% F present 0.4% MnO ₂ present
8	China clay ^c	17.50	17.48	-0.02	0.1% TiO ₂ present
9	China clay ^c	18.10	18.10	±0.00	
10	Beryl	11.40	11.36	-0.04	
11	Nepheline syenite ^c	13.00	13.00	±0.00	
12	Nepheline syenite ^c	11.10	11.10	±0.00	
14	Kyanite ^c	30.03	29.97	-0.06	
15	Glass sand	10.71	10.71	±0.00	
16	Potter's flint ^c	10.22	10.22	±0.00	
17	Aplite ^c	23.14	23.14	±0.00	
18	Spodumene	14.84	14.84	±0.00	
19	Amblygonite	11.03	11.06	+0.03	3.5% F present
20	Burned refractory ^c	37.67 ^b	37.69	+0.02	2.2% TiO ₂ present

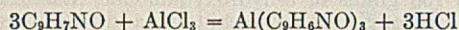
^a Values obtained in ordinary manner after removal of silica and recovery of nonvolatile matter.

^b National Bureau of Standards recommended value.

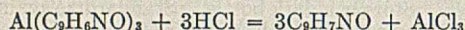
^c Correction for iron applied after precipitation of iron and alumina on basis of: Total ferric iron—0.1 mg. × 0.6 (10).

boiling for a few moments to coagulate the precipitated nickelic and ferric hydroxides. The solution is filtered through a Whatman No. 41H paper into a 250-ml. beaker. The nickel beaker is polished and the paper washed well with hot water. The paper is discarded.

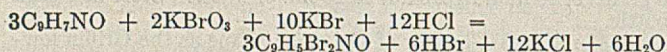
The filtrate is treated with 15 ml. of 8-hydroxyquinoline solution and the precipitate first formed is dissolved by stirring. Hydrochloric acid is now added, drop by drop, while stirring, through the stage of apparent complete precipitation, continuing until the precipitate is completely dissolved and the solution is distinctly acid. During this preliminary precipitation, observation of the color of the precipitate—ferric quinolate is distinctly green—will indicate whether the iron is present in sufficient quantity to necessitate preventing its precipitation. If this is required, sufficient hydroxylamine hydrochloride solution is added to effect reduction of ferric iron and the solution is heated to 80° to 90° C. An excess of α,α' -bipyridine hydrochloride or *o*-phenanthroline hydrochloride solution is added and the aluminum is precipitated by the addition of ammonium acetate buffer solution, 10 ml. in excess of that required for complete precipitation. The precipitation reaction is as follows:



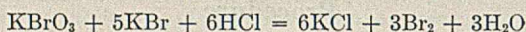
The suspension is coagulated by stirring if the iron quinolate reaction has been inhibited (the ferrous *o*-phenanthroline or ferrous bipyridine complex is dissociated by boiling) or by boiling if no effort was made to prevent the iron precipitation, and filtered through a Whatman No. 41H paper. The beaker is rinsed and the residue washed with cold water, the filtrate being discarded. The residue is dissolved directly from the paper with boiling 1 to 1 hydrochloric acid, catching the filtrate in the beaker in which the original precipitation was made. The paper is washed several times with boiling 5 per cent hydrochloric acid and discarded. The solution of the residue in hydrochloric acid again produces 8-hydroxyquinoline according to the reaction



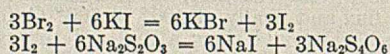
The filtrate containing the 8-hydroxyquinoline is made to 125 ml. with water and a measured excess of potassium bromate-bromide solution is added while stirring. The following reaction takes place:



The excess bromate-bromide reacts:



The addition of an excess (1 ml. is sufficient) of potassium iodide solution converts the free bromine to free iodine and this excess is measured by titration with sodium thiosulfate solution to a starch end point



This is calculated to percentage of alumina.

The results obtained by the procedure described are shown in Table I.

Discussion of Results

This procedure proved satisfactory for all the materials covered in Table I, though certain modifications were at times necessary.

The analyst can easily recognize the α,α' -bipyridine or *o*-phenanthroline requirements of the iron present as quinolate during the initial precipitation. Very little excess over the reagent required to satisfy the formula $Fe(C_{10}H_8N)_3Cl_2$ was necessary. In no case was any iron found in the aluminum quinolate residue.

Among the other metals which form quinolates and might interfere with the determination of aluminum as quinolate are titanium, zirconium, manganese, uranium, vanadium, and magnesium. Titanium and zirconium in the presence of iron will separate quantitatively as the hydroxide and will be found in the initial residue. Manganese separates as manganese dioxide in alkaline solution. Uranium and vanadium will probably be found with the aluminum, unless a second precipitation of the aluminum quinolate is made in a solution containing hydrogen peroxide. Magnesium forms the quinolate only in ammoniacal solution and no interference of this metal need be anticipated if the aluminum is precipitated under the conditions described.

Although the same basic procedure was used on all the samples covered in the study, certain changes in sample—flux ratio and the temperature cycle—were sometimes necessary.

FELDSPAR. The feldspars studied were all relatively free of iron. The sample (100 mg.) can be brought into solution with 1 gram of sodium hydroxide but hydrolysis of the aluminum hydroxide sometimes takes place unless more than 1.5 grams of the flux is used.

CORNWALL STONE. The Cornwall stone fused readily with 1.5 grams of sodium hydroxide when 100-mg. samples were used and the iron quinolate reaction was inhibited.

LEPIDOLITE. At least 2 grams of the flux are required for each 50 mg. of sample. Sample 7 proved high in iron and the reaction was inhibited.

CHINA CLAY. Because of its light, fluffy nature, the use of alcoholic sodium hydroxide was required. The sample and flux were moistened with alcohol and thoroughly dried before fusion. The flux-sample ratio was 40 to 1.

BERYL reacted in the same manner as the feldspar sample.

NEPHELINE SYENITE. Samples 11 and 12 represent unprocessed materials originally containing 4.72 and 3.70 per cent of ferric oxide, respectively. The ferric oxide contents of the alkali filtrates were 0.97 and 0.84 per cent, respectively, and necessitated the *o*-phenanthroline or bipyridine treatment. Because of the high alumina content at least 2 grams of flux are required for each 100 mg. of sample. Samples of this material which had been previously processed reacted like feldspar samples, though a greater amount of flux would prove advantageous in preventing hydrolysis.

KYANITE. A flux-sample ratio of 40 to 1 was required and it was found necessary to heat at a higher temperature and for a longer period to ensure complete decomposition.

GLASS SAND AND POTTER'S FLINT are similar in chemical composition. One gram of the sample and 5 grams of flux were used in either case.

APLITE was easily fused with 2 grams of flux per 100 mg. of sample.

SPODUMENE. A flux-sample ratio of 20 to 1 proved suitable.

AMBLYGONITE can be effectively brought into solution with a flux-sample ratio of 20 to 1.

BURNED REFRACTORY. A flux-sample ratio of 100 to 1 was required and it was found necessary to heat at a higher temperature for a longer period of time to ensure complete decomposition.

Conclusions

The determination of alumina as the quinolate in the presence of silica and iron is feasible and the method outlined offers definite advantages over those ordinarily employed for

this purpose. The method is economical because no platinum or extremely high temperatures are required and the reagent cost is low.

Acknowledgment

The author is indebted to Thomas C. Carson, Jr., for many helpful suggestions and analytical data.

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Determining Riboflavin in Dried Milk Products

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SEVERAL physico-chemical methods have been proposed for the determination of riboflavin, all of which may be grouped under one of three headings:

1. Preparation of a derivative whose solubility properties differ markedly from those of riboflavin, and determination of the color of the resulting solution (6).
2. Direct measurement of the color of riboflavin solutions (3).
3. Measurement of the fluorescence of riboflavin solutions (1).

By preparing a derivative, it is possible to separate the pigment from other colored substances which may accompany riboflavin in extracts of natural products. Thus, riboflavin is a water-soluble pigment, whereas lumiflavin is soluble in chloroform. However, the preparation of lumiflavin is accompanied by large losses (4, 6).

The direct measurement of the color of extracts of biological materials is subject to error due to the presence of other colored substances. This method may be somewhat refined (4) by controlled oxidation of these impurities. However, with more vigorous treatment, riboflavin is also destroyed, so that at times it is impossible to purify the extracts completely.

The determination of fluorescence is complicated by a number of factors, two of which are most obvious: The intensity of fluorescence is not a linear function of the concentration of riboflavin but passes through a maximum with increasing concentration (2). Extracts of biological materials contain blue (4) and white (7) fluorescing substances which interfere with the accurate determination of the green fluorescence due to riboflavin.

Since this vitamin has received increased attention during the past few years, it is apparent that a rapid, quantitative method of estimating riboflavin is still needed. Such a method was developed by the authors two years ago and has been in constant use since that time.

Principle of the Method

An extract of dried milk products is prepared and freed from unstable colored impurities by controlled oxidation. Light of the frequencies which are absorbed by riboflavin is passed

through the extract and the amount of absorption is determined with a photoelectric photometer. The riboflavin is then reduced to the leuco form and the measurement repeated to determine the amount of light absorbed by impurities. The concentration of riboflavin is calculated from the difference between these two readings.

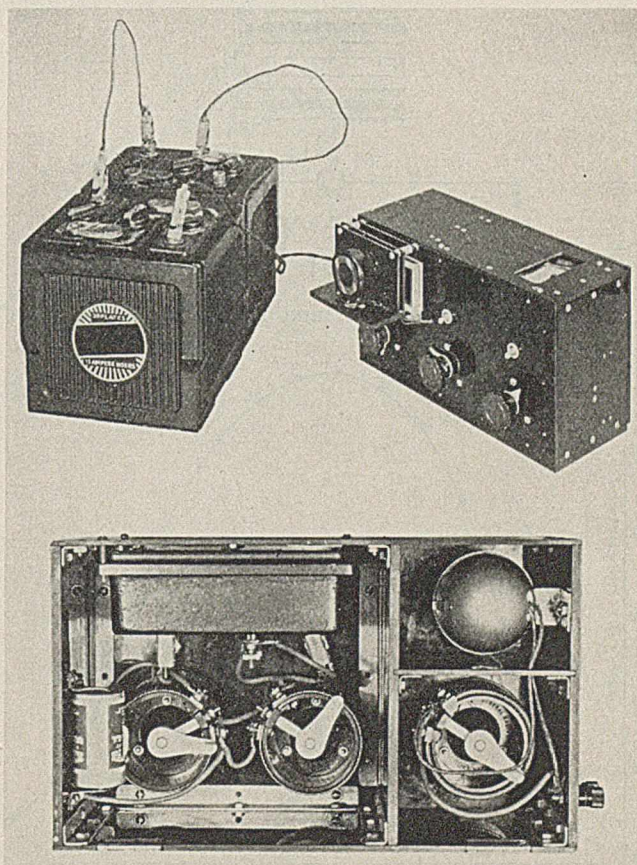


FIGURE 1. EXTERIOR AND INTERIOR VIEWS OF PHOTOMETER

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Apparatus

The photoelectric photometer, constructed for this work, employs a single photocell of the blocking-layer type. The zero-potential circuit, proposed by Wood (9) for use with these cells, has made it possible to obtain a current that is in exact proportion to the light intensity.

Views of the photometer are presented in Figure 1. The rear wall of the Bakelite box is placed on hinges, so that all parts are readily accessible.

A schematic wiring diagram is given in Figure 2. M is a microammeter with internal resistance of 150 ohms and critical damping resistance of 1000 ohms. B is a flashlight dry cell. K_1 and K_2 are contact keys, the former for controlling the dry cell and the latter for short-circuiting the photocell. R_1 , R_2 , and R_3 are potentiometer rheostats of 10,000, 1000, and 5 ohms' resistance, respectively. P is a blocking-layer photocell. D_1 , D_2 , D_3 , and D_4 are constant-aperture diaphragms which also serve to hold filters F_1 and F_2 in place. The light source, L , is a 9.6-volt flashlight bulb. Two 6-volt storage batteries, S , furnish the current for the light. A convenient type of absorption cell, A , is of 20-ml. capacity with a path length of 1 cm.

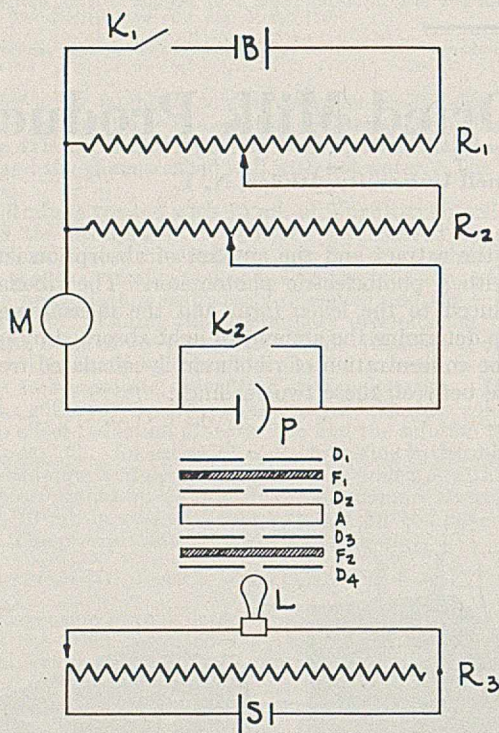


FIGURE 2. WIRING DIAGRAM

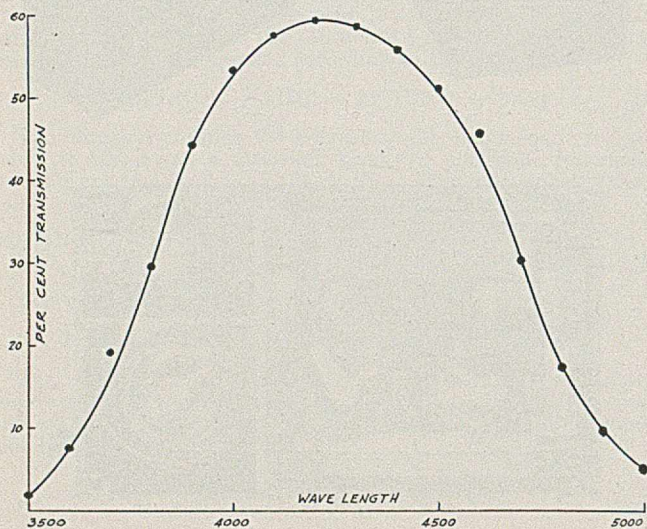


FIGURE 3. TRANSMISSION CURVE

The intense beam of light obtained from the flashlight bulb is rendered parallel by means of a reflector. Since the efficiency of the reflector has been found to decrease gradually over a period of months, 6, 8, 10, or 12 volts may be tapped off from the storage batteries to obtain the proper light intensity. The final adjustment is made by means of the 5-ohm rheostat.

Circular holes, 2.5 cm. (1 inch) in diameter, are cut in the partitions which support the filters, so that the beam striking the photocell is essentially parallel light perpendicular to its surface. In order to limit the illumination to light of the desired frequencies a set of H. R. lantern blue filters (Corning Glass Works, Corning, N. Y.) was selected. The transmission curve of these filters is given in Figure 3. One filter is placed between the light and the absorption cell, so that only light of the desired wave lengths is transmitted to the solution. This procedure reduces unnecessary heating of the solution. An identical filter is placed between the absorption cell and the photocell to filter out fluorescence caused by illumination of the sample. Even with low light intensities, certain materials, such as riboflavin, produce sufficient light to influence the reading. This precaution is particularly important when working with blue light, since the photocell is more sensitive to the longer wave lengths produced by fluorescing materials than to the original light.

Operation of the Photometer

An absorption cell filled with water, or other solvent to be used for the unknown, is placed in the path of the light. With switches K_1 and K_2 closed, the current from the dry cell is adjusted to give a full-scale deflection. This procedure enables one to predetermine the voltage required to balance the fall of potential across the ammeter when the photocell is producing a full-scale deflection. Switch K_2 is then opened and the light intensity is adjusted by means of the resistance in series with the lamp until a full-scale deflection is again obtained. If this has been properly done there will be no change in deflection upon opening and closing K_2 .

Another advantage in the present circuit is that with the photocell short-circuited, an unknown solution may be substituted for the solvent without causing the usual violent fluctuation of the ammeter. The current from the dry cell is reduced to approximately the reading expected and K_2 is opened. The direction in which the needle is deflected will indicate whether the applied voltage is too large or too small. K_2 is then closed and the current from the dry cell is modified accordingly. In this way the applied voltage is again adjusted to equal exactly the fall of potential across the am-

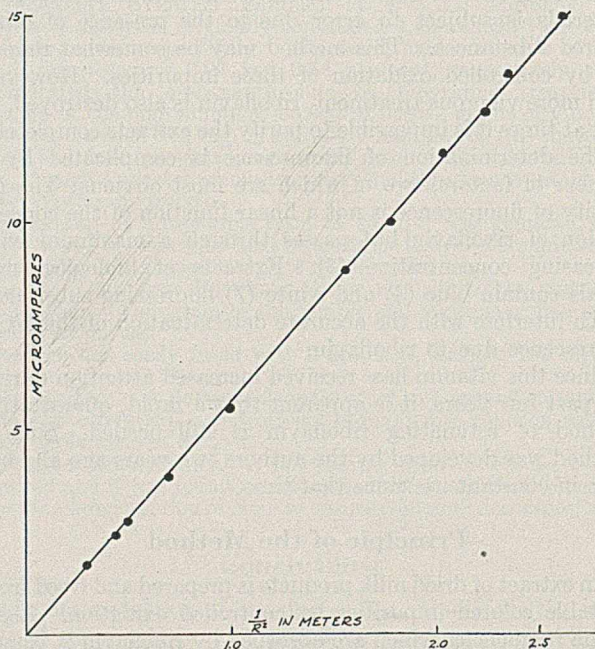


FIGURE 4. CALIBRATION CURVE

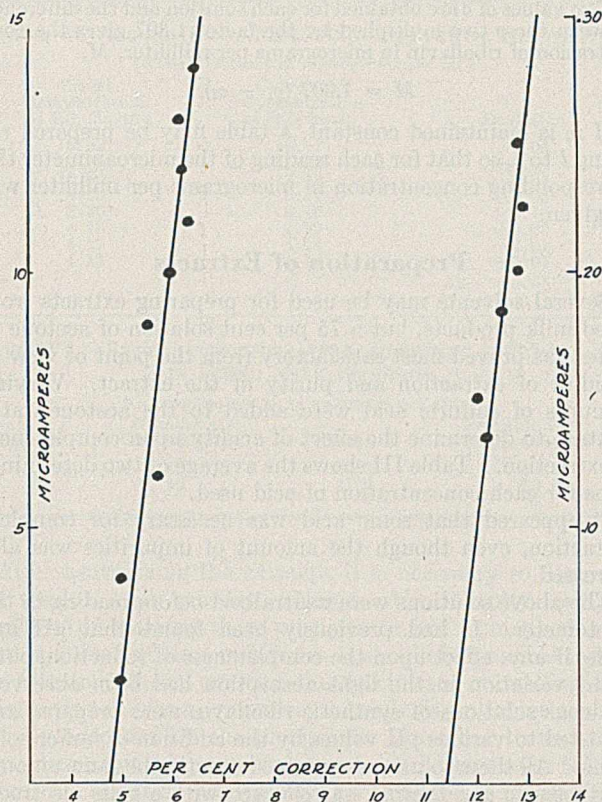


FIGURE 5. CORRECTION FACTOR

meter, so that no change in deflection is obtained upon opening or closing K_2 .

To demonstrate that this instrument would give a linear response to varying light intensities, observations were made as a light bulb was moved along an optical bench. The current obtained should be inversely proportional to the square of the distance separating the photocell from the light. These results are presented in Figure 4 and demonstrate that, with the zero-potential circuit, the current obtained from the photocell is a linear function of the light intensity.

The difference between these results and the readings which are obtained by connecting the photocell directly to the meter is demonstrated in Figure 5. Readings were made first in the usual manner and then with the zero-potential circuit and the difference between the two readings was calculated in per cent of the correct reading. The dependence of this correction upon the internal resistance of the meter is also shown in Figure 5, in which data are presented from two meters whose internal resistances were 150 and 450 ohms, respectively. Thus, the magnitude of the percentage correction varies both with the current produced and with the external resistance in the circuit.

Calibration of Photometer

A sample of pure synthetic riboflavin was obtained for calibrating the photometer. The absorption curve of this material is given in Figure 6. Its molecular extinction coefficient, 2.78×10^4 at 4450 Å., was found to be in essential

TABLE I. DENSITY OF SOLUTIONS OF SYNTHETIC RIBOFLAVIN

Concentration Micrograms/ml.	Log I_0/I Found	Log I_0/I Calculated	Per Cent Deviation
21.12	0.569	0.682	-16.6
16.90	0.460	0.543	-15.3
16.51	0.448	0.531	-15.6
13.52	0.373	0.437	-14.7
12.38	0.340	0.399	-14.8
10.82	0.298	0.351	-15.1
8.25	0.225	0.267	-15.7
6.49	0.178	0.209	-14.8
4.13	0.111	0.132	-15.9
3.89	0.106	0.126	-15.9
			Av. -15.4

agreement with that obtained by Kuhn (5). Since the response of the photometer depends not only upon the transmission of the filters but also upon the sensitivity of the photocell to different wave lengths and the energy distribution from the tungsten filament of the light bulb, the combined effect of these three factors has also been plotted in Figure 6. It is apparent that the final sensitivity of the instrument from 4000 to 5000 Å. is very nearly identical with the absorption curve of riboflavin.

In order to determine to what extent these filters would approximate the results obtained with monochromatic light, a sample of synthetic riboflavin was weighed on a microbalance and dissolved in distilled water. A series of dilutions was made up from this stock solution and the absorption of each solution was determined in the photometer. These results are given in Table I and demonstrate that the density ($\log I_0/I$), as determined with the photometer, is 15 per cent less than the theoretical value obtained with monochromatic light. The fact that solutions of riboflavin follow Beer's law is illustrated in Figure 7, in which these density values have been plotted against concentration.

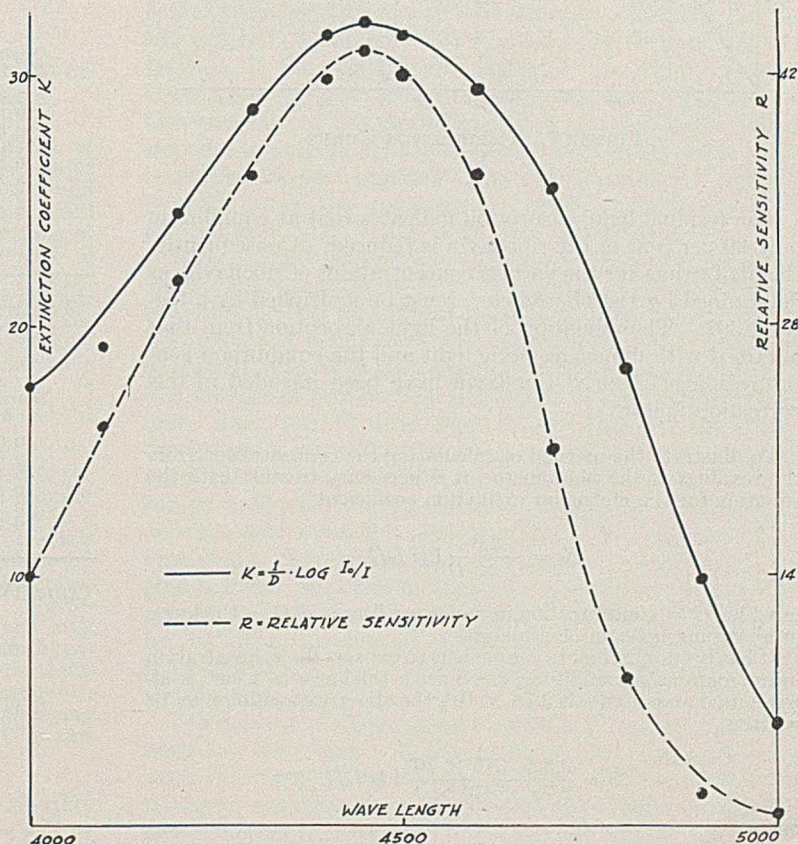


FIGURE 6. COMPARISON OF ABSORPTION CURVE WITH PHOTOMETER SENSITIVITY

For the purpose of reducing riboflavin to the leuco form, a solution is prepared by adding 5 per cent of sodium hyposulfite to a 2 per cent solution of sodium bicarbonate. This solution should be made up fresh each day and held in an ice bath while in use. One or two drops of this solution will reduce 20 ml. of a moderately concentrated solution of riboflavin. Riboflavin solutions of varying concentrations were reduced by this method, with the results shown in Table II.

Two values of c are obtained for each solution and the difference between these two multiplied by the factor 1.307 gives the concentration of riboflavin in micrograms per milliliter, M .

$$M = 1.307 (c_1 - c_2)$$

If I_0 is maintained constant, a table may be prepared relating I to c , so that for each reading of the microammeter the corresponding concentration in micrograms per milliliter will be given.

Preparation of Extracts

Several solvents may be used for preparing extracts from dried milk products, but a 75 per cent solution of acetone in water has proved most satisfactory from the point of view of rapidity of extraction and purity of the extract. Varying amounts of sulfuric acid were added to the acetone-water mixture to determine the effect of acidity upon completeness of extraction. Table III shows the average of two determinations for each concentration of acid used.

It appeared that some acid was necessary for complete extraction, even though the amount of impurities was also increased.

The above solutions were neutralized before reading in the photometer. It had previously been found that pH had little, if any, effect upon the completeness of reduction, but a slight variation in the light absorption had been observed. Aqueous solutions of synthetic riboflavin were prepared and adjusted to various pH values by the addition of buffer solutions. All these solutions contained exactly the same amount of riboflavin and the pH was checked with a glass electrode. Several observations were made of the light absorption at each pH value. It was found that the maximum absorption occurred at pH 7.0. Each observation was calculated in per cent of this maximum reading and recorded in Table IV.

TABLE II. REDUCTION OF SOLUTIONS OF SYNTHETIC RIBOFLAVIN WITH SODIUM HYPOSULFITE

(Concentrations calculated in micrograms per ml.)		
Original Reading	After Reduction	Per Cent Unreduced
13.92	1.32	9.5
10.59	1.03	9.7
10.59	1.08	10.2
7.05	0.69	9.8
6.98	0.74	10.6
3.53	0.32	9.1
3.53	0.36	10.2
3.53	0.36	10.2
		Av. 9.8

TABLE III. EFFECT OF ACIDITY UPON COMPLETENESS OF EXTRACTION FROM DRIED MILK POWDERS

(Concentrations in micrograms per ml.)		
Normality of Acid	Riboflavin	Impurities Calculated as Riboflavin
0.00	2.09	0.63
0.05	2.11	0.50
0.10	2.27	0.69
0.15	2.22	0.82
0.20	2.49	0.78
0.25	2.28	0.83

TABLE IV. EFFECT OF pH ON LIGHT ABSORPTION OF SOLUTIONS OF SYNTHETIC RIBOFLAVIN

pH	Per Cent of Maximum Absorption
1.0	96.0
2.0	98.7
3.0	96.4
4.0	96.8
5.0	96.5
6.0	98.4
7.0	100.0
8.0	97.5
9.0	95.3
10.0	94.5

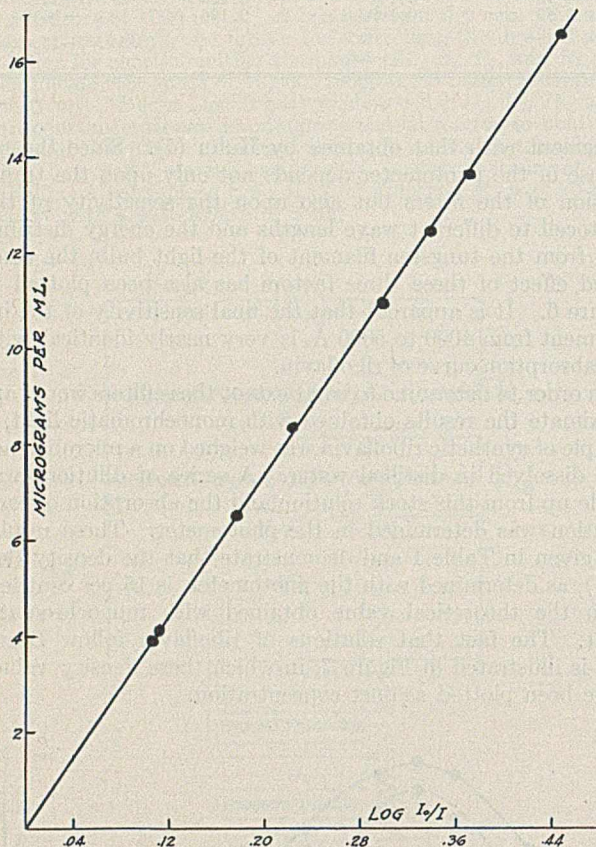


FIGURE 7. CALIBRATION CURVE

The residual light absorption indicates that at equilibrium only 90 per cent of the riboflavin is reduced. Consequently, the final values for the various concentrations of riboflavin, as determined by the photometer, must be multiplied by a factor 1.307. The deviation of the light absorption from that obtained with monochromatic light and the equilibrium concentration of reduced riboflavin have been included in this correction factor.

To illustrate the method of calculating the concentration from the readings of the photometer, it is necessary to start with the equation for the molecular extinction coefficient

$$K = \frac{2.3}{C \times D} \log I_0/I$$

in which C = concentration in moles per liter and D = thickness of absorbing layer in centimeters.

It has been found more convenient to express the concentration in micrograms per milliliter, c . Since a thickness of 1 cm. is always used and K equals 2.78×10^4 , the above expression may be written

$$c = \frac{2.3 \times 376 \times 10^3}{2.78 \times 10^4 \times 1} \log I_0/I$$

or

$$c = 31.1 \log I_0/I$$

TABLE V. ACCURACY OF MEASURING SYNTHETIC RIBOFLAVIN

(Concentrations in micrograms per ml.)		
Actual Concentration of Riboflavin	Calculated Concentration of Riboflavin	Deviation
16.52	16.47	-0.04
12.39	12.49	+0.10
12.39	12.43	+0.04
8.26	8.31	+0.05
8.26	8.16	-0.10
4.13	4.20	+0.07
4.13	4.14	+0.01
4.13	4.14	+0.01
Av.		±0.05

TABLE VI. REPRODUCIBILITY OF MEASUREMENT OF RIBOFLAVIN IN DRIED MILK PRODUCTS

(Concentrations in micrograms per gram)			
Sample	Number of Observations	Mean	Probable Error of Single Observation
1	12	29.8	±0.87
2	10	21.4	±0.62

After neutralizing the extracts, it is necessary to filter off the residual milk powder. Considerable difficulty was experienced in washing these precipitates free from riboflavin. With a known volume of solvent, it should be possible to use the filtrate without washing the precipitate or making the filtrate up to volume, provided that riboflavin is not preferentially adsorbed by the precipitate. In order to test this hypothesis, two 10-gram samples of dried whey were taken, a known amount of pure riboflavin was added to one sample, and both samples were then extracted, neutralized, and filtered. The light absorption of the filtrates was determined without washing the precipitates or making the filtrates up to volume.

Riboflavin Added Micrograms	Riboflavin Found Micrograms
0	300
160	460

Based upon these experiments, the following method of extracting riboflavin from dried milk products was devised.

An acid-acetone solution is prepared by making 750 ml. of acetone up to 1 liter with *N* sulfuric acid. A 15-gram sample of milk powder is weighed into a 250-ml. Erlenmeyer flask and 100 ml. of the acid-acetone solution are added. It has been found that certain impurities are readily destroyed by the addition of hydrogen peroxide. Therefore, depending upon the degree of caramelization exhibited by the sample, from 1 to 3 ml. of 30 per cent hydrogen peroxide are added to the sample. The flask is connected to a reflux condenser, and the solution is boiled for 20 minutes, although complete extraction is generally obtained by 15 minutes of refluxing. The flask is allowed to cool while still connected to the condenser to prevent loss of solvent. It is then tightly stoppered with a paraffined cork and placed in the refrigerator for 30 minutes.

The flask is then removed and the solution neutralized by the addition of 25 ml. of *N* sodium hydroxide. Depending upon the amount of hydrogen peroxide used, from 22 to 24 ml. of a buffer solution are added, which makes a total of 150 ml. of solvent for a 15-gram sample. For dried whey, a buffer solution of pH 6.8 is used. For dried skim milk and dried buttermilk a buffer solution of pH 4.6 is used. The flask is returned to the refrigerator for 30 minutes, after which the solution may be filtered. The filtrate represents a 10 per cent solution of the sample and may now be read in the photometer. The amount of sodium hypofluorite required to reduce this solution will vary with the completeness with which the hydrogen peroxide has been decomposed. Usually from 15 to 30 drops will be required, and a correction may be made for the increase in volume of extract caused by this addition.

In order to demonstrate the accuracy with which pure riboflavin may be measured in the photometer, reference will be made to the data presented in Table II regarding solutions of

synthetic riboflavin of known concentration. Table V presents the actual concentrations, as well as the concentrations calculated from the photometric reading by means of the correction factor.

The reproducibility with which riboflavin may be extracted from dried milk products and measured in the photometer is illustrated in Table VI, based upon observations of two dried milk products. The probable error of a single observation is less than 1 microgram per gram.

Discussion

The manufacturers of blocking-layer photocells frequently state that no external potential should be applied to these instruments. However, this invariably results when a photocell is connected directly to a meter. The use of the zero-potential circuit enables one to use the cell at all times in the manner in which it was intended to be used. It might have appeared possible to dispense with any correction had the magnitude of the correction been a constant percentage of the correct reading, since in absorption spectrophotometry only the ratios of two readings are used. However, the correction is not a constant factor and other considerations influence the choice of circuit.

The use of the zero-potential circuit greatly reduces the variability which is ordinarily found between photocells of the same manufacturer. This is true because the greatest source of variation between cells lies in the internal leakage resistance, which no longer influences the readings when the electrodes are maintained at zero potential. Also, changes in the internal leakage resistance due to differences in atmospheric conditions will no longer influence the readings. Consequently, identical readings are obtained by operators in widely separated laboratories.

By the proper selection of filters, the photometer may be used for many types of colorimetric measurements. One of the limitations in the use of filters instead of a monochromator is that strict proportionality between concentration and density ($\log I_0/I$) is obtained only over a limited range of concentrations, if the transmission curve of the filter does not exactly coincide with the absorption band of the pigment (δ). Consequently, if the photometer itself does not give a linear response to varying light intensities, a calibration curve must be plotted for each pigment. The curvature of this line will be made up of two components: (1) inherent in the photometer itself, and (2) due to a failure of the filters.

With the present instrument it is only necessary to determine over what range the filters fit the absorption curve of the unknown in order to make direct calculation of the concentration from the readings of the photometer. This becomes important when it is necessary to determine one component of a mixture of pigments by the difference between two readings. This may be done when it is possible to destroy or decolorize the pigment being measured. It is obvious that an error will be involved if such a pair of readings is applied to a curved calibration curve, since the error due to the filters depends only upon the concentration of the pigment being measured. However, if the photometer is constructed to give a linear response to varying light intensities and the concentration of the unknown pigment is within the limits set by the filters, the change in light absorption upon removing the pigment will be a linear function of the concentration of the pigment.

The method of determining riboflavin which has been presented in this paper will give accurate and reproducible results when applied to well prepared samples of dried milk products, but when the milk powders have been caramelized, or when other feedstuffs are present in the sample, it is necessary to subject the extracts to further purification.

In several cases the method has been applied to milk products which were also assayed for riboflavin by means of chicks. The chick assays upon six samples gave an average value of 21.2 micrograms per gram. An average value of 21.5 micrograms per gram was obtained by the photometric test. The method presented in this paper is preferred because of its rapidity, and because biological assays may be complicated at times by the presence or absence of nutritional factors which are not as yet clearly defined.

The absolute accuracy of this method depends upon the purity of the sample of synthetic riboflavin used as a standard. Several samples of crystalline riboflavin were obtained from different sources at the time of standardizing the method and none of these appeared to be quite so pure as the sample used. This conclusion was based both upon the light absorption and the completeness of reduction. Since that time an occasional sample has been encountered which appeared to be reduced to a greater extent than the standard. When a satisfactory standard of reference has been obtained it may be necessary to alter slightly the above correction factor for calculating the concentration of riboflavin.

When other instruments are set up according to the above directions the same correction factor may be used for calculating riboflavin concentration. Some care must be taken to assure that exactly 90 per cent reduction of the riboflavin is obtained. Variation in the completeness of reduction may occur through incomplete removal of the peroxide or decomposition of the hyposulfite solution.

Conclusions

A photometer has been described which utilizes a zero-potential circuit in connection with a blocking-layer photocell. Such an instrument gives a linear response to varying light intensities, and offers advantages in regard to proper use of the photocell, reproducibility of results, and application to certain spectrophotometric measurements.

Riboflavin may be extracted from dried milk products by refluxing with a dilute solution of acid in 75 per cent acetone.

Certain unstable colored impurities may be destroyed by including from 1 to 3 per cent of hydrogen peroxide in the solvent. The resulting solution, after neutralizing and filtering, may be used for quantitative measurements since riboflavin is not preferentially adsorbed by the precipitated milk solids. By reduction with sodium hyposulfite, 90 per cent of the color of riboflavin is removed. Observation of the light absorption before and after reduction enables one to calculate the concentration of the solution to ± 0.05 microgram per ml.

Acknowledgments

The sample of synthetic riboflavin was kindly supplied by Richard Kuhn of the Kaiser Wilhelm Institut, Heidelberg, Germany. The transmission curve of the filters and the absorption curve of riboflavin were measured by A. E. Sidwell, Jr., of the University of Chicago. The work reported in this paper was made possible by the establishment at Cornell University of a special temporary fellowship, by the Kraft-Phenix Cheese Corporation of Chicago, Ill.

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PRESENTED before the Division of Agricultural and Food Chemistry at the Mid-West Meeting of the American Chemical Society, Omaha, Nebr. This paper was compiled largely from a thesis presented to the faculty of Cornell University in partial fulfillment of the requirement for the degree of doctor of philosophy, February, 1937, and was part of an experiment station project under the direction of L. C. Norris.

Boron Determination in Soils and Plants

Using the Quinalizarin Reaction

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IN HUMID regions, boron often exists in soils largely as tourmaline, a boroaluminum silicate which is not readily available. The available boron, probably existing often largely as the calcium salt, is usually present in humid regions in amounts of less than one part per million. In plant tissue, boron is often present to the extent of 25 parts per million, and since it is now recognized as an essential plant nutrient, its determination in both soils and plants assumes considerable importance. Methods used in the past for making these determinations have not been very satisfactory in some respects, and the present paper reports the results of an attempt at improvement.

In the past, the turmeric paper test described by Bertrand and Agulhon (1) has often been used in the determination of small amounts of boron. As the turmeric paper is not very sensitive to small differences in amounts of boron, and difficulties are encountered in making accurate comparisons with the standard, the method leaves much to be desired. The spectroscopic method has recently been used by a number of

investigators (3, 6) to determine the boron content of plant materials. It seems to give fairly good results, but requires rather elaborate equipment and is time-consuming. The titrimetric procedure, involving the use of polyhydroxy alcohols and discussed in detail by several workers (8, 11), is not well adapted for the rapid determination of the small amounts sometimes encountered in soil and plant analysis, because the method involves distillation which is time-consuming, and because traces of buffering substances which are difficult to remove may seriously interfere with the results.

Quinalizarin Reaction

It has been known for a considerable number of years that the addition of boric acid to many of the hydroxyanthraquinones in concentrated sulfuric acid will cause a marked color change which may be used for the identification of these quinones. Recently this color change was applied to the determination of boron. For this purpose, Feigl and Krumholz (5) tried purpurin, alizarin S, and quinalizarin. Quinaliz-

arin (1,2,5,8-tetrahydroxyanthraquinone) was found to be the most sensitive, changing from a pink color to a bluish hue with increasing concentrations of boron. Scharrer and Gottschall (8) found quinalizarin to be better than purpurin. These investigators found that fluorides, nitrates, dichromates, and other oxidizing compounds interfere in the test by turning the solution colorless.

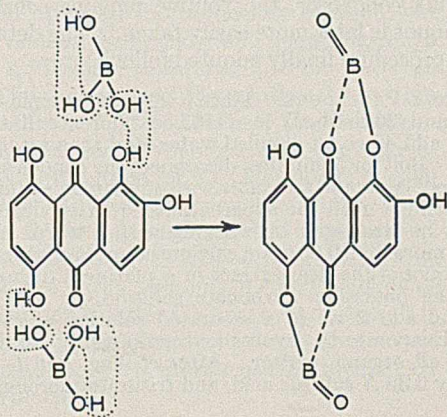
The authors found that if there are over 500 parts per million of water-soluble fluorides in soils, or 5000 parts per million of total fluorides in soils or plant tissue, the color on which the procedures to be described depend will be partially destroyed. Because of the insolubility of calcium fluoride, the presence of appreciable amounts of water-soluble fluorides in soils is rare. The small amounts ordinarily present in soils and plants do not interfere. Larger amounts will destroy it entirely. When necessary, fluorides are removed easily by precipitation with thorium chloride and subsequent filtration.

The only oxidizing substance found in soils and plants that interferes in the quinalizarin reaction is nitrate. In the procedures to be described, this is removed by ignition.

The only known substance (7, 10) that is similar to boron in causing a color change with quinalizarin is germanic acid. Poluektov (7) found that this also forms complex acids with polyhydroxy alcohols and thus enters into an alkalimetric titration like boric acid. In tests made by the authors, it was found that it takes over 200 times as much germanium as boron to produce a certain color change in the quinalizarin reaction. This means that in the procedures described below there would have to be over 20 parts per million of water-soluble germanium in soils, and over 200 parts per million of total germanium in soils or plant tissue, to produce a visible color change. Since the occurrence of germanium in soils and plants is rare, and since it takes considerable amounts of germanium to produce a visible color change, interference by germanium in the quinalizarin test for boron in soils and plants appears to be remote.

An all-important factor in the determination of boron by means of the quinalizarin reaction is the concentration of sulfuric acid in which the color is developed. Smith (9) found that at least 44 per cent by weight of sulfuric acid must be present in the solution in order to produce a visible color change due to boron. He found that the sensitivity or color change increases as the concentration of sulfuric acid increases until about 93 per cent of acid by weight is present; beyond this the sensitivity decreases. Smith indicates that the method is sensitive to 0.001 mg. of boric acid. He applied the method to the determination of boron in alloys.

The interaction of boric acid with quinalizarin is undoubtedly very similar to its reaction with dihydroxyanthraquinones, which form chelated compounds with boric acid as shown by Feigl and Krumholz (5). In accordance with this, the quinalizarin reaction with boric acid takes place:



Four molecules of water are split off and a chelated ring forms as indicated. A similar reaction takes place between boric acid and open-chain polyhydroxy alcohols (4). The reaction given reflects the necessity of the presence of concentrated sulfuric acid to aid in splitting off the water and prevent the reaction from reversing.

For the determination of easily soluble boron in soils, the writers first tried the turmeric paper test but the results obtained were not entirely satisfactory. The quinalizarin color reaction was then applied to both soil and plant analysis, and a satisfactory procedure was developed, the details of which follow.

Application of Quinalizarin Reaction

In the application of the quinalizarin reaction, it was found that a concentration of about 93 per cent by weight of sulfuric acid in the final test solution, obtained by adding 98.5 per cent acid by weight, gives the most satisfactory results for small amounts. With this concentration and method of comparison used, it was possible to distinguish 0.0001 mg. of boron from a blank test.

Nitrates in amounts usually found in soil solutions may be reduced and removed by adding potassium carbonate and powdered aluminum to the soil extract and evaporating. Later it was found that evaporation and gentle ignition in the presence of potassium carbonate or calcium hydroxide were more convenient and fully effective in removing the nitrates ordinarily found in soil extracts. This also destroys organic matter which otherwise produces a brown color and interferes with the test color.

Organic matter and many other substances may also be separated by distilling the boron as the methyl ester of boric acid. To do this, the soil extract is evaporated to dryness and then taken up in 85 per cent phosphoric acid and some absolute methyl alcohol. The solution is placed in a distilling flask (boron-free glass) and the methyl borate distilled into an aqueous solution of potassium carbonate. The presence of a small amount of free water prevents the complete distillation of the boron. This may be guarded against by the addition of a small amount of phosphoric anhydride.

The distillation and subsequent evaporation necessary to obtain the desired concentration were time-consuming and laborious; hence, the simple ignition procedure was adopted to destroy both nitrates and organic matter, which appear to be the only interfering substances ordinarily present in soil extracts. When 0.1 gram of potassium carbonate was added to the soil extract, and the extract was then evaporated to dryness and gently ignited, there was no loss of boron, while nitrates and organic matter were destroyed. Platinum containers must be used when the ignition is made with potassium carbonate, since this reagent attacks both quartz and porcelain ware. Calcium hydroxide may be used in place of potassium carbonate, making possible evaporation and ignition in porcelain or quartz dishes. The ignition and distillation procedures gave identical results when applied to synthetic solutions and soil extracts.

Standard color solutions for the colorimetric comparisons may be prepared by adding the appropriate amounts of the reagents to varying amounts of boron as in testing unknown solutions. These standards are permanent if kept in stoppered comparison tubes, so as to prevent absorption of water. Since it is necessary to remove the stoppers to make comparisons most advantageously, absorption of water cannot be entirely prevented; hence, these standards usually deteriorate in the course of a week or so, depending upon amount of exposure. The color comparisons can, of course, also be made by means of a photoelectric colorimeter.

TABLE I. PREPARATION OF COLOR STANDARDS

No.	Boron Present in Color Standards Mg.	Amounts Required to Produce Corresponding Standards	
		Distilled water Cc.	stock solutions B and C Cc.
1	0.0000	1.00	0.00
2	0.0002	0.80	0.20 (C)
3	0.0004	0.60	0.40 (C)
4	0.0006	0.40	0.60 (C)
5	0.0008	0.20	0.80 (C)
6	0.0010	0.00	1.00 (C)
7	0.0015	0.85	0.15 (B)
8	0.0020	0.80	0.20 (B)
9	0.0025	0.75	0.25 (B)
10	0.0030	0.70	0.30 (B)
11	0.0035	0.65	0.35 (B)
12	0.0040	0.60	0.40 (B)

Preparation of Reagents

SULFURIC ACID, 98.5 PER CENT BY WEIGHT. Although the strength of sulfuric acid may vary from 98 to 99 per cent by weight, for small amounts of boron it is very important to keep it within these limits, and this requires accurate work in its preparation and great care in its use and storage. The acid is prepared by mixing ordinary concentrated sulfuric acid with fuming sulfuric acid. To facilitate the calculations involved in determining the proportion of each to be mixed, strengths are best expressed in terms of sulfur trioxide rather than sulfuric acid. Accordingly, the desired 98.5 per cent sulfuric acid becomes 80.4 per cent sulfur trioxide. The proportion of each to be mixed varies, of course, with the strengths of the acids. The concentrated acid usually contains about 95 per cent of sulfuric acid by weight, and the fuming 20 to 30 per cent of free sulfur trioxide. The exact strength in each case is determined by weighing out 2 grams or more of the acid in a 25-cc. weighing bottle, and after diluting, titrating with 1.0 *N* sodium hydroxide. In the case of the concentrated sulfuric acid, the weighing bottle with contents is placed in a beaker of water, and, after mixing, the acid is titrated. In the case of the fuming acid, the weighing bottle containing the acid is dropped into a second 100-cc. weighing bottle containing about 30 cc. of water. As the bottle is dropped, the cover of the 25-cc. bottle is removed, and then the cover of the larger bottle is quickly replaced. After standing overnight or until fuming has entirely ceased, the two weighing bottles with covers removed are placed in a liter beaker containing 300 to 400 cc. of water. It is advisable to place the cover of the larger bottle in the beaker also, to prevent any loss of acid which might be on the cover. After mixing, the acid is titrated. The strengths in terms of sulfur trioxide are then calculated by means of the following formula:

$$\frac{\text{cc. NaOH titration} \times \text{normality} \times 0.04003}{\text{weight of concentrated or fuming H}_2\text{SO}_4} \times 100 = \text{percentage by weight of SO}_2 \text{ in each case}$$

After the strengths of the acids have been determined in terms of sulfur trioxide, the proportion of each to be mixed to make 100 grams of 98.5 per cent sulfuric acid, which contains 80.40 per cent of sulfur trioxide, is calculated as follows:

Let x = grams of concentrated sulfuric acid needed
 $100 - x$ = grams of fuming sulfuric acid needed
 Let a = strength of concentrated sulfuric acid in terms of sulfur trioxide expressed decimally
 b = strength of fuming sulfuric acid in terms of sulfur trioxide expressed decimally
 Then, $ax + b(100 - x) = 80.40$, and x is solved for
EXAMPLE. Strength of concentrated sulfuric acid found by titration was 77.78 per cent sulfur trioxide. Strength of fuming sulfuric acid found by titration was 87.41 per cent sulfur trioxide. Substituting these values in the formula above and solving for x , there is obtained:
 $0.7778x + 0.8741(100 - x) = 80.40$
 $0.0963x = 7.01$
 $x = 72.79$ grams, amount of concentrated acid needed
 $100 - 72.79 = 27.21$ grams of fuming acid needed in making 100 grams of desired mixture containing 98.5 per cent H_2SO_4

QUINALIZARIN SOLUTION. Dissolve 0.01 gram of quinalizarin in 100 cc. of strong sulfuric acid made by diluting 9 volumes of 98.5 per cent by weight sulfuric acid with one volume of water. Store in a glass-stoppered bottle.

SULFURIC ACID, APPROXIMATELY 0.36 *N*. Dilute 5 cc. of 95 to 96 per cent by weight sulfuric acid to 500 cc. with distilled water.

SULFURIC ACID, APPROXIMATELY 4 *N*. Dilute 50 cc. of 95 to 96 per cent by weight sulfuric acid to 450 cc. with distilled water.

CALCIUM HYDROXIDE, SATURATED SOLUTION. Add 5 to 10 grams of calcium hydroxide to 500 cc. of distilled water. Shake well and allow to settle.

POTASSIUM CARBONATE SOLUTION. Dissolve 40 grams of anhydrous potassium carbonate in 100 cc. of distilled water. Five drops of this contain about 0.1 gram of potassium carbonate.

Preparation of Color Standards

Dissolve 2.8578 grams of boric acid in 1000 cc. of distilled water. This solution contains 0.5 mg. of boron per cc. and serves as the primary (A) base stock solution. Prepare a second (B) stock solution containing 0.01 mg. of boron per cc. by diluting 20 cc. of the primary base stock solution to 1000 cc. with distilled water, and a third (C) stock solution containing 0.001 mg. of boron per cc. by diluting 100 cc. of the second stock solution to 1000 cc.

Transfer varying amounts of the second stock solution for amounts above 0.001 mg. of boron, and of the third stock solution for the others, to boron-free test tubes or glass vials. Glass vials approximately 20×100 mm. are convenient and satisfactory for this purpose, and the amounts of boron given in Table I produce a satisfactory range of colors. In order to have the correct concentration of acid when the color is developed, it is necessary to have exactly 1 cc. of boric acid solution in each vial. This is conveniently done by dispensing the boron stock solutions from burets, and then adding water from another buret to bring the volume to 1 cc. in each case. Now, add 9 cc. of the 98.5 per cent sulfuric acid, or other acid, to be used with the unknown test solution. Stopper the vials, cool, and add 0.5 cc. of the quinalizarin solution. After 15 minutes or more the color becomes fully developed and the standards are ready for use. These colors are permanent if the vials are kept stoppered to prevent absorption of water.

Determination of Available Boron in Soils

Most of the available boron in soils is undoubtedly water-soluble. It was found that when a known amount of soluble boron in the form of boric acid was added to several soils free of water-soluble boron, and the soils were then dried, the added boron could be completely recovered by adding water, boiling for 5 minutes, and then filtering. The addition of hot water, followed by shaking for 30 minutes and then filtering, did not result in complete recovery. Complete recovery may be effected by extraction with dilute acid. This procedure, however, raises complications when calcareous soils are encountered, because of the difficulty of regulating the acidity. Furthermore, tests made with acid extractions of calcareous soils indicated that the results thus obtained often do not correlate well with crop indications of the boron status. After numerous tests, refluxing of the soil-water suspension for 5 minutes appeared to be the best procedure. More boron was extracted by refluxing from 5 to 10 minutes than either for shorter or longer periods. Boiling with a reflux condenser, the volume remains constant, and thus an aliquot is later more easily taken. The details of the analytical procedure finally adopted follow.

ANALYTICAL PROCEDURE. Place a 20-gram sample of the soil (air-dried and 20-meshed) in a 125-cc. Florence flask (boron-free glass), add 40 cc. of distilled water, and then attach a reflux condenser. Boil for 5 minutes, disconnect the condenser, stopper the flask, cool contents, and filter with suction on a Büchner funnel or centrifuge until the supernatant liquid is clear. Clarification may be facilitated by working with a warm solution or adding not more than 0.05 gram calcium chloride dihydrate. Place a 20-cc. aliquot of the clear extract in a platinum dish and add 5 drops of the potassium carbonate solution, or in a porcelain crucible and add 2 cc. of a saturated solution of calcium hydroxide. Evaporate to dryness and ignite gently to destroy nitrates and all organic matter. After cooling, add 5 cc. of approximately 0.36 *N* sulfuric acid, and triturate thoroughly with a policeman.

TABLE II. AVAILABLE OR WATER-SOLUBLE BORON FOUND IN FIELD TEST PLATS FERTILIZED WITH BORAX

Source of Sample	Type of Soil	pH	Boron Applied as Borax	Available Boron Found in Soils		
				A	B	Av.
Plat 10	Clyde silt loam	8.0	0.00	0.50	0.50	0.50
Plat 11	Clyde silt loam	7.8	0.55	1.00	1.00	1.00
Plat 12	Clyde silt loam	7.6	1.10	1.00	1.10	1.05
Plat 13	Clyde silt loam	7.5	1.65	1.30	1.30	1.30
Plat 14	Clyde silt loam	7.4	2.20	2.60	2.50	2.55
Plat 15	Clyde silt loam	7.8	2.75	2.30	2.20	2.25
Plat 100	Clyde sandy loam	8.0	0.00	0.40	0.45	0.43
Plat 101	Clyde sandy loam	8.0	1.10	0.90	0.95	0.93
Plat 23	Poygan clay loam	6.3	0.00	0.45	0.47	0.46
Plat 24	Poygan clay loam	6.4	1.10	1.50	1.40	1.45

TABLE III. BORON FOUND IN SOILS TO WHICH KNOWN AMOUNTS OF BORON AS BORIC ACID HAD BEEN ADDED

Type of Soil	Boron Added	Total Boron Found			Av.
		A	B	C	
Plainfield sand	0	2.0	2.0	2.0	2.0
	10	12.0	12.0	12.0	12.0
	20	22.0	22.0	22.0	22.0
Poygan clay loam	0	20.0	20.0	20.0	20.0
	10	28.0	30.0	30.0	29.3
	20	40.0	40.0	38.0	39.3
Miami silt loam	0	28.0	28.0	29.0	28.3
	10	38.0	38.0	38.0	38.0
	20	46.0	48.0	48.0	47.3

Place a 1-cc. aliquot of this solution in a comparison vial (20 × 100 mm.), add 9 cc. of the 98.5 per cent sulfuric acid, stopper the vial, and cool. After cooling, add 0.5 cc. of the quinalizarin reagent to the test solution and mix thoroughly by gently whirling the vial. Allow to stand for at least 15 minutes, and then determine the boron content by comparing with a set of standards. The final comparison is best made by removing the stopper from the standard and making a vertical observation against a white background, as is usually done in colorimetric comparisons.

TYPICAL RESULTS. Table II gives the amounts of available boron found in a number of field plats variously fertilized with borax. The borax was applied several months in advance of the soil sampling, and hence, some losses due to leaching undoubtedly occurred, which probably explains in part the incomplete recovery of the added boron. Some of the boron may also have become fixed in unavailable form. With these limitations in mind, the correlation between amounts of boron added and found appears satisfactory. Additional results, recently obtained and to be published elsewhere, correlate well with the crop indications.

Determination of Total Boron in Soils

In determining the total boron content of soils by means of the fusion method, it is necessary to use a high proportion of sodium carbonate to soil. Treatment of the melt obtained with water alone will bring all the boron into solution, but is inconvenient, being slow and requiring much water. Addition of sulfuric acid to the water, so that the final reaction of the solution falls within the pH range of 5.5 to 6, hastens the disintegration of the melt and leaves most of the sesquioxides and silica in insoluble form. Addition of alcohol up to 60 to 70 per cent by volume at this point serves to throw down most of the large amount of sodium sulfate which has been formed. This leaves all the boron and only a small amount of salts in solution. After the final evaporation, it is necessary to ignite because of the small amount of nonvolatile organic matter usually introduced with the alcohol.

ANALYTICAL PROCEDURE. Fuse 0.5 gram of soil with 3 grams of anhydrous sodium carbonate in a platinum crucible. Cool and place the crucible in a 250-cc. beaker containing about 50 cc. of distilled water. Place a cover glass on the beaker and add approximately 4 *N* sulfuric acid from time to time until the melt has disintegrated and the solution has a reaction in the range of pH 5.5 to 6.0. Transfer the resulting solution to a 500-cc. volumetric flask. Wash the beaker and crucible several times with distilled water and add the washings to the flask. The total volume of solution should now not exceed 150 cc. Add methyl or ethyl alcohol to the flask until a volume of 500 cc. is reached and mix the contents thoroughly. Filter the solution or centrifuge until the supernatant liquid is clear.

Place a 400-cc. aliquot of the clear solution in a 500-cc. beaker (boron-free glass) and add 100 to 150 cc. of distilled water to prevent subsequent precipitation. Add potassium carbonate until the solution is alkaline, evaporate to a small volume, and transfer to a platinum dish. Evaporate to dryness and ignite to destroy organic matter. After cooling, add 4 cc. of approximately 0.36 *N* sulfuric acid, and triturate thoroughly with a policeman. Place a 1-cc. aliquot of this solution in a comparison vial, add 9 cc. of the 98.5 per cent sulfuric acid, stopper the vial, and cool. After cooling, add 0.5 cc. of the quinalizarin reagent to the test solution and mix thoroughly by gently whirling the vial. Allow to stand at least 15 minutes before comparing with standards.

RESULTS. Table III gives results obtained in the analysis of several soils with this procedure. To some samples of these soils known amounts of boron had been added; recovery of the added boron was satisfactory. A Bureau of Standards sample of glass containing 0.22 per cent of boron was analyzed with the procedure just described, and six determinations gave 0.21, 0.23, 0.225, 0.22, 0.20, and 0.225 per cent of boron. The quinalizarin colorimetric procedure, because of its extreme sensitivity, is thus well adapted for the determination of the small amounts of boron usually found in soils, plants, and many other substances. When materials containing several per cent of boron are analyzed, the sample involved in the final comparison is so small that any slight error at this stage becomes multiplied many times in the calculations that follow. The titrimetric procedure may thus be better adapted to analyses involving high contents of boron. It is possible that a better adaptation of the colorimetric procedure for high contents might be attained by lowering the acidity of the test solution so as to require a larger amount of boron to effect a certain change in color intensity. The color standards whether for direct comparison or photoelectric calibration would, of course, need to be developed with the lower acidity also.

TABLE IV. BORON FOUND IN PLANT TISSUE ASHED WITH AND WITHOUT POTASSIUM CARBONATE

Nature of Plant Tissue	Nature of Culture	Amounts of Boron— (Oven-dry basis)					
		Ashed without K ₂ CO ₃			Ashed with K ₂ CO ₃		
		A	B	Av.	A	B	Av.
Alfalfa, leaves and stems	Quartz pot culture	36.0	36.0	36.0	35.0	34.0	34.5
Lettuce, leaves	Quartz pot culture	14.0	14.0	14.0	13.0	13.5	13.25
Beet, red, leaves and petioles	Field culture	30.0	30.0	30.0	28.0	30.0	29.0
Beet, red, leaves and petioles	Field culture	26.0	24.0	25.0	25.0	24.0	24.5
Beet, red, roots	Field culture	9.0	9.0	9.0	8.0	8.0	8.0
Red cabbage, leaves	Field culture	11.5	12.0	11.75	12.0	12.0	12.0
Okra, leaves	Field culture	26.0	26.0	26.0	26.0	24.0	25.0
Tomato, leaves and petioles	Field culture	40.0	38.0	39.0	40.0	40.0	40.0
Shortleaf pine seedlings	Field culture	9.0	9.0	9.0	9.0	9.0	9.0
Horsetail, leaves	Field culture	11.0	11.0	11.0	11.0	11.0	11.0
Cattail, leaves	Field culture	12.0	12.0	12.0	11.0	12.0	11.5
Lemon, fruit pulp	Field culture	11.5	11.5	11.5	11.5	11.5	11.5
Lemon, fruit juice ^a	Field culture	0.3	0.3	0.3	0.3	0.3	0.3

^a Results calculated on basis of juice, not oven-dried residue.

TABLE V. BORON CONTENT OF VARIOUS PLANTS GROWN ON SAME SOIL

Species of Plant	Portion of Plant	Amounts of Boron Found		
		A	B	Av.
		P. p. m.		
Sweet corn	Leaves	4	4	4.0
Kale	Leaves and petioles	12	13	12.5
Leek	Leaves and bulb	12	13	12.5
Red cabbage	Leaves	12	12	12.0
Green beans	Leaves and stems	15	15	15.0
Carrot	Roots	15	14	14.5
Carrot	Leaves and petioles	16	18	17.0
White cabbage	Leaves	16	18	17.0
Okra	Leaves	18	18	18.0
Tomato	Leaves	40	39	39.5

Determination of Total Boron in Plants

ANALYTICAL PROCEDURE. Place a 0.25- to 0.50-gram sample of plant tissue (oven-dried and ground) in a platinum crucible or porcelain evaporating dish and ignite gently to a white or gray ash. After cooling add 5 cc. of approximately 0.36 *N* sulfuric acid and triturate with a policeman. After settling, place a 1-cc. aliquot of the clear supernatant liquid in a comparison vial, add 9 cc. of the 98.5 per cent sulfuric acid, stopper the vial, and cool. Then add 0.5 cc. of quinalizarin solution, stopper, and mix well by whirling gently. Allow to stand at least 15 minutes and then determine the boron content by comparing with a set of standards.

RESULTS. The results of analyses with and without the addition of potassium carbonate prior to ashing, given in Table IV, show that it is not necessary to add a base to the plant tissue to prevent loss of boron on ashing. Several plants—namely, shortleaf pine, horsetail, and cattail—which have a preponderance of acidic constituents in their ash, were ignited with and without the addition of a base; no significant differences in boron content were noted. A sample of lemon juice was neutralized, and this juice, together with a sample to which no base had been added, was evaporated to dryness and ignited. The results show that even with this very acid juice the boron suffers no loss on ignition. Other workers (2, 6) have obtained similar results. Apparently practically all plant tissue and juices release sufficient bases from organic combination when ignition takes place to hold the boron as a nonvolatile borate.

Table V gives the boron content of a number of species of plants grown on the same soil. The amounts found correspond well with the amounts found by other investigators (2). The boron content of corn leaves is much lower than that of many vegetables. Vegetables, fruits, truck crops, and legumes seem to suffer sooner from a lack of boron than the grasses, and this seems to be correlated with the boron content of these plants.

Table VI gives the boron content of a number of species of plants and the available boron in the culture mediums in which the plants were grown. These data show that there is some correlation between the amount of available boron in the culture medium and that found in plant tissue grown thereon. This is especially true in the case of quartz cultures in which the amount of available boron can be closely controlled.

Precautions

Since many c. p. chemicals contain appreciable amounts of boron, it is essential that all chemicals used in the determination of boron be tested for freedom from this element. Pyrex glass contains about 11 per cent of boric oxide and may cause serious contamination if used in this determination. Kavalier or other boron-free glassware should be used. All reagents should be stored in containers made of boron-free glass.

Common soft-glass bottles are usually satisfactory. Great care must be exercised in measuring the 1-cc. aliquot of the unknown to which are added the reagents for color development, because an error of 1 drop in this measurement may cause an error of from 5 to 10 per cent in the final result, through its influence on the final acid concentration in the mixture. The 98.5 per cent sulfuric acid reagent and the quinalizarin solution should be stored in bottles equipped with tightly fitting glass stoppers to prevent absorption of water, and these bottles should not be left unstoppered. The 98.5 per cent sulfuric acid solution should be tested from time to time, so as to be certain of its strength. Pipets, burets, and all other measuring instruments should be calibrated.

TABLE VI. BORON FOUND IN PLANT TISSUE PRODUCED UNDER VARIOUS CONDITIONS

Species of Plant	Portion of Plant	Nature of Culture	Amounts of Boron	
			Available in culture medium	Total in plant tissue
			P. p. m.	
Beets, red	Leaves and petioles	Field culture	0.43	14.5
	Roots	Field culture	0.43	6.0
	Leaves and petioles	Field culture	0.93	24.0
	Roots	Field culture	0.93	9.0
Lettuce	Leaves	Quartz pot culture	0.00	3.8
			0.25	7.0
			0.50	10.3
			1.00	13.5
			2.00	10.5
Alfalfa	Leaves and stems	Quartz pot culture	0.00	8.0
			0.50	33.5
			1.00	41.5
			2.00	51.0

Summary

By means of the quinalizarin color reaction it is possible with proper control of the acidity to detect 0.0001 mg. of boron. This reaction was found to be well adapted for the detection and colorimetric determination of the small amounts of boron found in soils, plants, and many other materials. Interfering substances ordinarily present in soil extracts and plant tissues are easily removed by a simple ignition procedure.

The available boron of soils is extracted by refluxing with water for 5 minutes. An aliquot of the filtered extract is then made alkaline and evaporated to dryness. The residue is ignited to destroy organic matter and nitrates, and then taken up with dilute acid, after which the quinalizarin colorimetric test for boron is applied.

In the determination of total boron in soils and silicates, a sodium carbonate fusion is made and the resulting melt is dissolved at pH 5.5 to 6.0 so as to leave insoluble most of the silica and sesquioxides. The bulk of the sulfate is thrown out of solution by the addition of alcohol. The filtered solution is made alkaline, evaporated to dryness, and the residue ignited. After dissolving in dilute acid, the quinalizarin test is applied.

In the determination of the total boron of plants, the plant tissue is ignited to a gray ash which is taken up with dilute acid. The quinalizarin test is then applied to some of the clarified extract.

A considerable number of soils, some of which had been fertilized with borax, and plants variously fertilized with boron were analyzed. The results obtained were fairly consistent with the treatments given. The test for available boron of soils appears to be well adapted for determining the boron status of a soil.

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Volume-Shape Factor of Particulate Matter

Probable Errors in the Computation

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THE volume of an aggregate of irregularly shaped particles can be determined from their average diameter by the simple relation

$$V = \delta ND^3 \quad (1)$$

where δ is the volume-shape factor (for spheres = $\pi/6$), N the number of particles, and D the average diameter. The value of δ is always less than $\pi/6 = 0.5236$, and depends upon characteristics of the material, its mode of cleavage during crushing, and perhaps to some extent the particle size. The shape factor of small particles should approximate that for a sphere as their particle size decreases, particularly since the methods used for the measurement of their diameters frequently does not distinguish any dominant irregularities. However this may be, there are data to warrant the assumption that a representative sample of an aggregate has a fairly constant shape factor (1, 3). This paper furnishes experimental evidence of the variation in shape factor for crushed quartz particles between the range of 10- and 40-mesh Bureau of Standards calibrated screens.

Two sieve sizes were used in the experiments described—namely, those particles passing a 10-mesh sieve and retained on a 20-mesh, and those passing this and retained on a 40-

mesh. The sizes of sieve openings were: 10-mesh, 0.2 cm.; 20-mesh, 0.084 cm.; and 40-mesh, 0.042. The sample was taken from the sieve and stored in a glass container. When aliquots were to be counted and weighed in accordance with the procedure discussed below, the total sample was placed upon a sheet of glazed paper and quartered.

The particles were carefully sieved and average diameters of upwards to 1000 particles in each sample determined by the technique developed by Hatch and Choate (3). The diameters of particles having an average volume were 0.214 and 0.089 cm., indicating rather significant differences from the sieve sizes given above. (Logarithmic mean sizes of the corresponding samples are 0.19 ± 0.05 and 0.089 ± 0.05 cm.) These differences have been discussed at length by Hatch in a paper on the determination of average size from sieve analysis (2). No significant variations were found in the particle sizes for each of the two samples for aliquots less than 1000 but more than 100 particles. Thus, the average diameters may be assumed as representative of the aggregates in question.

To determine the variation in shape factor, portions of each sample were weighed and counted and the average weight of each particle was calculated. This procedure

TABLE I. AVERAGE WEIGHT OF QUARTZ PARTICLES PASSING 10-MESH AND RETAINED ON 20-MESH

Weight of Particles Grams	Number of Particles	Average Weight per Particle		
		10^{-3} gram	10^{-5}	10^{-10}
0.5572	128	435	- 2	4
0.9811	187	525	+88	7744
2.0674	443	467	+30	900
2.0979	495	424	-13	169
2.4995	472	530	+93	8649
4.0260	947	425	-12	144
4.1882	930	450	+13	169
4.2124	1028	409	-28	784
4.2855	1090	393	-44	1936
4.8081	1284	374	-63	3969
5.5026	1111	495	+58	3364
6.0034	1521	395	-42	1764
9.1695	2084	440	+ 3	9
17.6686	4170	424	-13	169
17.6951	4139	428	- 9	81
17.8439	4455	401	-36	1296
17.8482	4396	406	-31	961

$$\text{Average weight per particle} = \frac{7421}{17} \times 10^{-5} = 437 \times 10^{-5} \text{ gram.}$$

$$\text{Standard deviation} = \pm \sqrt{\frac{31,112}{17}} \times 10^{-5} = \pm 428 \times 10^{-6} \text{ gram.}$$

$$\text{Probable error of any determination} = 428 \times 10^{-6} \times 0.6745 = 301 \times 10^{-6} = 6.9\%.$$

TABLE II. AVERAGE WEIGHT OF QUARTZ PARTICLES PASSING 20-MESH AND RETAINED ON 40-MESH

Weight of Sample Grams	Number of Particles	Average Weight per Particle		
		10^{-6} gram	10^{-6}	10^{-12}
0.8805	3500	252	-18	324
0.8844	3500	253	-17	289
0.9366	3600	260	-10	100
0.9889	3700	267	- 3	9
0.9962	3600	276	+ 6	36
1.0014	4150	241	-29	841
1.0037	3700	271	+ 1	1
1.0069	3800	265	- 5	25
1.0310	3600	287	+17	289
1.0324	4230	245	-25	625
1.0413	3800	274	+ 4	16
1.0504	3679	285	+15	225
1.0920	4228	258	-12	144
1.1125	3529	300	+30	900
1.1171	3800	294	+24	576
2.0010	6817	293	+23	529
2.1484	8030	268	- 2	4
3.0039	11328	265	- 5	25
4.0189	14779	272	+ 2	4
5.0043	18129	276	+ 6	36

$$\text{Average weight per particle} = \frac{5402}{20} \times 10^{-6} = 270 \times 10^{-6} \text{ gram.}$$

$$\text{Standard deviation} = \pm \sqrt{\frac{4998}{20}} \times 10^{-6} = \pm 50 \times 10^{-6}.$$

$$\text{Probable error} = 50 \times 10^{-6} \times 0.6745 = 33.7 \times 10^{-6} = 12.5\%.$$

is in itself a measure of the shape factor, since the weight of the particles is obtained from Equation 1 by multiplying by the density

$$W = \delta \rho ND^3 \quad (2)$$

Since D and ρ are known and may be considered constant,

$$\delta = K \frac{W}{N} \quad (3)$$

Hence, the shape factor is as accurate as the average weight of the particles constituting the sample. Average particle weights were obtained by counting all particles from several portions of each sample, and reweighing. The results of this procedure are given in Tables I and II.

The tables show that the average particle weights as determined above are not constant. Nor does there appear to be any trend toward constancy, even when large numbers of particles are counted. The probable errors of individual samples are 6.9 and 12.5 per cent, which may be considered as being rather large.

The shape factors calculated from Equation 1 for the samples in question are 0.17 and 0.15, respectively (taking $\rho = 2.65$). These values correspond with the shape factors determined by Hatch and Choate for very small particles (3). These investigators obtained volume-shape factors as low as 0.14 for particles of the order of 10 microns (0.001 cm.). Actual particle counts, however, were not made, but obtained by an indirect procedure.

Without question, the size distribution of the aggregate is most important. A small number of large particles can alter the ratio of W/N although no significant change in the size distribution, and hence the average diameter, is apparent. The volume of a particle is as the cube of the diameter. Hence, a single particle of an aggregate with a diameter 10 times that of the smallest has a volume (and mass) equivalent to 1000 particles of the latter. This fact undoubtedly accounts for the differences in average particle weights. (As a matter of experiment, the extremes of size-frequency plots vary from sample to sample without materially affecting the average or the median sizes.) It is questionable whether a size-weight distribution for calculating average diameters would yield better results since, as Hatch has pointed out, summation curves by weight and count will plot as parallel lines on logarithmic probability paper (2). It may be concluded, therefore, that the best value of the volume-shape factor is that determined from the average particle weights of several aliquots of the aggregate, each including a large number of particles.

Acknowledgment

The authors desire to acknowledge the assistance given them by Richard W. Schayer.

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Judging Adhesiveness of Bitumen to Silica Sand

A Comparison of Mixing Method and Wash Test

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THE fact that a certain parallelism exists between wetting and adhesiveness phenomena has tempted a number of investigators to apply the principles and methods of flotation to the problem of testing and improving the adhesiveness of bitumen to mineral aggregate. The inherent theoretical and practical limitations of such an undertaking have been outlined previously (4). Here it need only be mentioned that while wetting phenomena are usually instantaneous, the development of maximum adhesion between a mineral surface and bituminous material is a function of time. A recent contribution applying flotation principles to the problem of bitumen adhesion is the work of McLeod (2), who while using activators and wetting agents assumes that the ease of covering wet Ottawa sand with asphaltic bitumen is a measure of adhesiveness. Since the ease and speed with which the mixing test by McLeod can be performed would represent an advantage over the more lengthy procedure of the commonly used wash test (1, 3, 5), it appeared desirable to compare results of these two tests on the same set of samples.

Experimental Procedure

The testing procedures used were the mixing method as reported by McLeod and the wash test with a machine previously described by the authors (5). For the experiments on mixing, Ottawa sand was thoroughly washed with distilled water and dried, and the fraction passing No. 8 and retained on a No. 40 sieve was employed. Two asphaltic materials having the properties given in Table I, and 0.05 N solutions of sodium oleate and activator salts, respectively, were used for the tests. A

preliminary experiment showed that various MC and SC oils had different mixing efficiencies with the sand in the presence of water. However, the addition of from 0.2 to 0.5 cc. of 0.05 N sodium oleate solution to any of the asphalt-sand-water mixtures resulted in a complete stripping of the coated sand and the balling up of the asphalt into numerous particles. The addition, thereafter, of the activator solutions had different effects, varying with the concentration and type of compound. The mixtures for testing adhesion were made in the following manner:

Thirty grams of sand and 1 gram of asphaltic material were thoroughly mixed in the presence of water, enough water being added to cover the sand and the excess drained off.

TABLE I. PROPERTIES OF ASPHALTS

	Slow-Curing Road Oil	
	No. 1	No. 2
Saybolt-Furol viscosity at 122° F., sec.	171	327
Specific gravity, 60° F./60° F.	1.0402	0.9766
Flash point, ° F.	240	260
Oliensis	Positive	Negative
Residue, %	71.8	73.2
Penetration of residue at 77° F., 100 grams, 5 sec.	86	85
Ductility of residue at 77° F., cm.	101	150+
Loss on heating 50 grams 5 hours at 325° F., %	9.92	8.36

Varying degrees of coverage were obtained.

One-half cubic centimeter of 0.05 N sodium oleate was added and the mixture stirred; complete stripping occurred.

The activator solution was added in 0.1-cc. increments with thorough mixing after each addition until the sand was uniformly coated or until it was seen that no coating would be accomplished.

The adhesion data and mixing observations are given in Table II. The adhesiveness tests were made after the samples had cured in air 3 days and then stood in water one day.

Results

Asphalt 1, when mixed with the sand in the absence of water, wetting, and activating agents gave a very good coating, and after the same curing period as described above gave the best possible adhesion. Mixed in the presence of water, the mixing was still very good, whereas after the same curing period as above the asphalt stripped at 60° C., being not affected at 30° and 45° C., which can be called fair adhesion.

Asphalt 2, when mixed with the sand in the absence of water, wetting, and activating agents, gave a poor mix and poor adhesion. Mixing and adhesion were also very poor when this asphalt and sand were mixed in the presence of water.

These two asphalts provide the two extremes for observation—e. g., very good and very poor adhesion. The data in Table II indicate a similarity in the results of mixing observations and adhesion tests if both activator and wetting agent are used. There is, however, no exact correlation, because several mixes which were very good on the basis of efficient mixing gave poor adhesion. These cases are magnesium chloride and silver nitrate with asphalt 2. These two salts also lowered the adhesion of asphalt 1.

Since the wetting agent (sodium oleate) in the absence of an activator caused the complete stripping of the asphalts from the sand, the effect of omitting the wetting agent and adding only the so-called activator solution was investigated. Results of these experiments are given in Table III. In this case, the amount of water present is more important than where the activator and wetting agent are used.

These tests indicate that the salt solutions which are bad with soap are also bad when soap is not used. However, some of the salt solutions which have a good effect in connection with soap do not give good results when soap is not used. Lead nitrate appeared to be the best reagent in regard to both mixing and adhesion results, although potassium aluminum sulfate was almost as good. Calcium chloride, magnesium chloride, and silver nitrate were generally not good with regard to adhesion, although magnesium chloride and silver nitrate tended in proper amounts to give good mixing (with soap). Ferric chloride used without soap gave

TABLE III. RESULTS USING ACTIVATOR AND OMITTING WETTING AGENT

Reagent	0.05 N		Mix	30°	45°	60°	70°
	Cc.	H ₂ O Cc.					
Asphalt 1							
Co(NO ₃) ₂	1	7	Medium	1	1	1	1
	5	3	Medium	1	1	1	1
MgCl ₂	1	7	Poor	1	1	1	1
	5	3	Poor	1	1	1	1
CaCl ₂	1	7	Poor	1	1	1	1
	5	3	Poor	1	1	1	3
BaCl ₂	1	7	Medium	1	1	1	3
	5	3	Medium	1	1	1	1
Pb(NO ₃) ₂	1	7	Very good	1	1	1	1
	5	3	Very good	1	1	1	1
KAl(SO ₄) ₂	1	7	Very good	1	1	1	1
	5	3	Very good	1	1	1	1
CuSO ₄	1	7	Very good	1	1	1	1
	5	3	Very good	1	1	1	3
FeCl ₃	1	7	Good	1	1	1	1
	5	3	Poor	1	1	1	1
AgNO ₃	1	7	Good	1	1	2	3
	5	3	Good	1	1	1	1
ZnSO ₄	1	7	Very good	1	1	3	..
	5	3	Very good	1	1	3	..
None	..	None	Very good	1	1	1	1
	..	8	Very good	1	1	3	..
Asphalt 2							
CaCl ₂	1	7	Poor	3
	5	3	Poor	3
KAl(SO ₄) ₂	1	7	Poor	1	3
	5	3	Poor	1	1	1	1
Pb(NO ₃) ₂	1	7	Poor	1	1	1	3
	5	3	Poor	1	1	1	1
BaCl ₂	1	7	Poor	1	1	1	1
	5	3	Poor	1	1	1	1
AgNO ₃	1	7	Poor	3
	5	3	Poor	1	3
FeCl ₃	1	7	Poor	1	1	1	1
	5	3	Poor	1	1	1	1
CuSO ₄	1	7	Poor	3
	5	3	Good	1	3
MgCl ₂	1	7	Poor	3
	5	3	Poor	3
Co(NO ₃) ₂	1	7	Poor	3
	5	3	Poor	3
None	..	8	Poor	3
	..	None	Poor	3

TABLE II. MIXING AND ADHESION DATA

(0.5 cc. of sodium oleate used)

Soap	Salt	Salt Solution, Cc.	Mix	Adhesion Value ^a			
				30°	45°	60°	70°
Asphalt 1							
1	Co(NO ₃) ₂	0.2	Uniform	1	1	1	1
2	KAl(SO ₄) ₂	0.1	Uniform	1	1	1	1
3	FeCl ₃	0.1	Uniform	1	1	1	1
4	Pb(NO ₃) ₂	0.1	Uniform	1	1	1	1
5	ZnSO ₄	0.2	Uniform	1	1	1	1
6	AgNO ₃	0.9	Uniform	1	1	1	3
7	CaCl ₂	0.5	Uniform	1	1	3	3
8	BaCl ₂	0.2	Uniform	1	1	1	1
9	MgCl ₂	0.5	Uniform	1	1	1	3
10	CuSO ₄	0.2	Uniform	1	1	1	1
Asphalt 2							
Coating							
1	Co(NO ₃) ₂	0.2	Uniform	1	1	1	1
2	KAl(SO ₄) ₂	0.1	Uniform	1	1	1	1
3	FeCl ₃	0.3	Uniform	1	1	1	1
4	Pb(NO ₃) ₂	0.1	Uniform	1	1	1	1
5	ZnSO ₄	0.2	Uniform	1	1	1	1
6	AgNO ₃	1.2	Poor until 1.2 cc. were added	3
7	CaCl ₂	2.1	Very poor	3
8	BaCl ₂	0.1	Good	1	1	1	1
9	MgCl ₂	0.5	Poor until 5 cc. were added	2	3
10	CuSO ₄	0.1	Uniform	1	1	1	1

^a 1 = no stripping, 2 = 25% or less stripped, 3 = more than 25% stripped.

good mixing when added in very small quantities, but as the quantity was increased the mixing efficiency decreased. However, any particles which were coated in the presence of ferric chloride gave very good adhesion.

Conclusions

The ease of covering a sand with a bituminous material by means of simple mixing may be a general indication, but is not a dependable measure of the adhesion relationship between the bitumen and the sand.

The best agreement of the mixing test data with those from the wash test occurs where wetting agents are used in conjunction with aluminum, iron, and lead ions—i. e., with those ions which are generally recognized as favorably affecting adhesion of bitumen to aggregate. For such cases and where the aggregate has to be coated in the wet condition with the help of soap-type activators the McLeod test appears to have some merit.

The mixing test cannot be substituted for the wash test if definite information on the adhesion relationship between bitumen and aggregate is desired.

Acknowledgment

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A Table for Ebulliometers

For Use with Alcoholic Liquids Containing Solid Matter

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THE ebulliometer is used in many industrial plants such as wineries, breweries, and distilleries for rapid determination of the alcohol content of wine, beer, mash, and distilled liquors. The tables or scales which accompany the ebulliometers are constructed for liquids containing either no solid matter or one rather definite quantity of solids such as would be found in beer or light wine.

Since the variety of alcoholic liquids which the ebulliometer may be called upon to analyze is rather large and may cover solid contents of considerable range, a table which takes into account such solid matter may be of value.

To obtain data with which to construct such a table, mixtures of 192-proof alcohol, sucrose, and distilled water were prepared containing approximately 1, 3, 5, 7, 9, and 11 per cent of alcohol by volume accurately determined by a pycnometer, and 0, 2, 4, 6, 8, and 10 grams of sucrose per 100 ml.—a total of 36 solutions. Dilutions were made with a buret and a volumetric flask which were checked against each other, and the liquids were measured at 20° C.

Each solution was tested, simultaneously, in a Juerst and a Salleron ebulliometer. The thermometer used with the Juerst was manufactured by the Taylor Instrument Company, was graduated the same as the Juerst thermometers, and was certified by the National Bureau of Standards. The thermometer used with the Salleron was the one furnished with the ebulliometer,

and although it had not been certified by the National Bureau of Standards, it agreed very closely with the Taylor instrument. In a third ebulliometer, water was kept boiling continuously to detect any change in boiling point during the periods of testing.

Before each series of tests, the ebulliometers were cleaned by boiling with a strong solution of alkali to remove the film of solid matter which collects inside the boiler after only a few tests are made and which causes bumping. Solutions containing more than 10 grams per 100 ml. of sucrose were not used because of the tendency of liquids of high-solid content to bump. Such liquids are best analyzed by diluting the samples. Even after thorough washing of the boiler, the first boiling point of water was usually different from the succeeding ones; therefore the boiling point of water was obtained by boiling successive portions of water until several duplicate results were obtained.

The boiler was then thoroughly rinsed with the alcoholic liquid to be tested, but in spite of that fact, the boiling point of the solution was affected by the previous presence of water and the first reading had to be discarded, as it was usually high, corresponding to a percentage of alcohol which was too low.

The boiling point of each solution was determined at least six times with each ebulliometer. The readings were made by observing the thermometers every 15 seconds from the time boiling started until the mercury remained constant during several observations. The point at which the mercury was

TABLE I. BOILING POINT DIFFERENCES, JUERST EBULLIOMETER

Alcohol %	Grams of Sucrose per 100 Ml. of Solution					
	0	2	4	6	8	10
0.9585	0.95	0.92	0.89	0.86	0.83	0.82
2.8755	2.68	2.68	2.69	2.72	2.67	2.70
4.7925	4.20	4.23	4.22	4.25	4.27	4.30
6.7095	5.60	5.65	5.66	5.71	5.73	5.76
8.6265	6.83	6.91	6.92	7.02	7.07	7.12
10.5435	7.96	8.02	8.12	8.19	8.17	8.27

TABLE II. ADJUSTED RESULTS USED IN CONSTRUCTING A TABLE, JUERST EBULLIOMETER

Alcohol %	Grams of Sucrose per 100 Ml. of Solution					
	0	2	4	6	8	10
0.9585	0.95	0.92	0.89	0.86	0.83	0.80
2.8755	2.68	2.68	2.68	2.68	2.68	2.68
4.7925	4.20	4.22	4.24	4.26	4.28	4.30
6.7095	5.60	5.63	5.66	5.69	5.72	5.75
8.6265	6.82	6.88	6.94	7.00	7.06	7.12
10.5435	7.96	8.02	8.08	8.14	8.20	8.26

TABLE III. BOILING POINT DIFFERENCES, SALLERON EBULLIOMETER

Alcohol %	Grams of Sucrose per 100 Ml. of Solution					
	0	2	4	6	8	10
0.9585	0.94	0.92	0.89	0.88	0.85	0.84
2.8755	2.68	2.68	2.69	2.70	2.70	2.70
4.7925	4.20	4.23	4.26	4.29	4.31	4.35
6.7095	5.60	5.65	5.66	5.71	5.77	5.81
8.6255	6.84	6.90	6.92	6.96	7.06	7.10
10.5435	7.96	8.02	8.09	8.16	8.17	8.26

TABLE IV. ADJUSTED RESULTS, SALLERON EBULLIOMETER

Alcohol %	Grams of Sucrose per 100 Ml. of Solution					
	0	2	4	6	8	10
0.9585	0.94	0.92	0.90	0.88	0.86	0.84
2.8755	2.69	2.69	2.69	2.69	2.69	2.69
4.7925	4.20	4.23	4.26	4.29	4.32	4.35
6.7095	5.60	5.64	5.68	5.72	5.76	5.80
8.6255	6.84	6.89	6.94	6.99	7.04	7.09
10.5435	7.95	8.01	8.07	8.13	8.19	8.25

TABLE V. READINGS WITH JUERST EBULLIOMETER

Sucrose Grams/100 ml.	Alcohol % by volume	Boiling Point Difference Found	High	Low	Variation
0	0.9585	0.95	0.95	0.94	0.01
0	2.8755	2.68	2.69	2.67	0.02
0	4.7925	4.20	4.22	4.19	0.03
0	6.7095	5.61	5.63	5.59	0.04
0	8.6265	6.83	6.85	6.82	0.03
0	10.5435	7.98	7.99	7.97	0.02
2	0.9585	0.92	0.93	0.92	0.01
2	2.8755	2.68	2.69	2.67	0.02
2	4.7925	4.23	4.24	4.22	0.02
2	6.7095	5.65	5.68	5.62	0.06
2	8.6265	6.91	6.93	6.87	0.06
2	10.5435	8.04	8.05	8.03	0.02
4	0.9585	0.89	0.89	0.90	0.01
4	2.8755	2.69	2.70	2.67	0.03
4	4.7925	4.22	4.25	4.20	0.05
4	6.7095	5.66	5.70	5.63	0.07
4	8.6265	6.92	6.93	6.90	0.05
4	10.5435	8.12	8.14	8.10	0.04
6	0.9585	0.86	0.87	0.85	0.02
6	2.8755	2.72	2.73	2.70	0.03
6	4.7925	4.25	4.27	4.22	0.05
6	6.7095	5.71	5.74	5.68	0.06
6	8.6265	7.02	7.04	7.00	0.04
6	10.5435	8.19	8.21	8.18	0.03
8	0.9585	0.83	0.84	0.82	0.02
8	2.8755	2.67	2.70	2.63	0.07
8	4.7925	4.27	4.30	4.23	0.07
8	6.7095	5.73	5.76	5.71	0.05
8	8.6265	7.07	7.11	7.03	0.08
8	10.5435	8.17	8.18	8.13	0.05
10	0.9585	0.82	0.84	0.81	0.03
10	2.8755	2.70	2.71	2.68	0.03
10	4.7925	4.30	4.32	4.26	0.06
10	6.7095	5.76	5.78	5.73	0.05
10	8.6265	7.12	7.15	7.10	0.05
10	10.5435	8.27	8.29	8.24	0.05

TABLE VI. FORMULAS FOR CALCULATING EBULLIOMETER TABLE

(Figures in first column represent grams of sucrose per 100 ml. of solution)

0	$y = 0.98469x + 0.03004x^2 + 0.00158x^3$
2	$y = 1.00618x + 0.02088x^2 + 0.00219x^3$
4	$y = 1.03436x + 0.00997x^2 + 0.00291x^3$
6	$y = 1.05861x + 0.00072x^2 + 0.00348x^3$
8	$y = 1.08209x - 0.00817x^2 + 0.00402x^3$
10	$y = 1.11577x - 0.02054x^2 + 0.00484x^3$

stationary for the longest period was recorded and the total of such readings averaged. Readings were estimated to hundredths of a degree on each thermometer. Results obtained, in boiling point differences, are shown in Table I for the Juerst and in Table III for the Salleron. Since these figures were slightly irregular, they were adjusted so that when plotted, smooth curves would be obtained. Table II shows the adjusted results used for constructing the table for

the Juerst ebulliometer. The adjusted results for the Salleron, shown in Table IV, were so nearly like the corresponding ones for the Juerst that one table was considered satisfactory for both ebulliometers. Table V shows the highest and lowest readings obtained for each solution and proves that consistent results can be obtained by using care in all of the manipulations. The variations in individual readings on the Salleron thermometer were larger than those on the Juerst because the

TABLE VII. TABLE TO ACCOMPANY EBULLIOMETER

(Per cent of alcohol by volume corresponding to differences in boiling points for quantities of sucrose between 0 and 10 grams per 100 ml.)

Boiling Point Difference	Grams of Sucrose per 100 Ml. of Solution					Boiling Point Difference	Grams of Sucrose per 100 Ml. of Solution						
	0	2	4	6	8		10	0	2	4	6	8	10
	<i>Per cent by volume</i>						<i>Per cent by volume</i>						
-0.15	0.01	0.0	3.95	4.45	4.43	4.42	4.41	4.40	4.39
-0.10	0.04	0.04	4.00	4.52	4.50	4.48	4.47	4.46	4.45
-0.05	0.06	0.09							
0.0	0.0	0.02	0.05	0.08	0.11	0.14	4.05	4.59	4.57	4.55	4.53	4.52	4.51
0.05	0.05	0.07	0.10	0.13	0.16	0.19	4.10	4.65	4.63	4.61	4.59	4.58	4.57
0.10	0.10	0.12	0.15	0.18	0.21	0.24	4.15	4.72	4.70	4.68	4.66	4.64	4.63
0.15	0.15	0.17	0.19	0.22	0.25	0.28	4.20	4.78	4.76	4.74	4.72	4.70	4.68
0.20	0.20	0.22	0.24	0.27	0.30	0.33	4.25	4.85	4.83	4.81	4.78	4.76	4.74
0.25	0.25	0.27	0.29	0.32	0.35	0.38	4.30	4.91	4.89	4.87	4.84	4.82	4.80
0.30	0.30	0.32	0.34	0.37	0.40	0.43	4.35	4.98	4.96	4.93	4.90	4.88	4.86
0.35	0.35	0.37	0.39	0.41	0.44	0.47	4.40	5.04	5.02	4.99	4.96	4.94	4.92
0.40	0.40	0.42	0.44	0.46	0.49	0.52	4.45	5.11	5.08	5.05	5.02	5.00	4.98
0.45	0.45	0.47	0.49	0.51	0.54	0.57	4.50	5.18	5.15	5.12	5.09	5.06	5.04
0.50	0.50	0.52	0.54	0.56	0.59	0.62	4.55	5.25	5.22	5.19	5.16	5.13	5.10
0.55	0.55	0.57	0.59	0.61	0.64	0.67	4.60	5.32	5.28	5.25	5.22	5.19	5.16
0.60	0.60	0.62	0.64	0.66	0.69	0.72	4.65	5.38	5.34	5.31	5.28	5.25	5.22
0.65	0.65	0.67	0.69	0.71	0.73	0.76	4.70	5.45	5.41	5.38	5.35	5.32	5.29
0.70	0.70	0.72	0.74	0.76	0.78	0.81	4.75	5.52	5.48	5.45	5.41	5.38	5.35
0.75	0.75	0.77	0.79	0.81	0.83	0.86	4.80	5.59	5.55	5.52	5.48	5.45	5.42
0.80	0.81	0.83	0.85	0.87	0.89	0.91	4.85	5.66	5.62	5.59	5.55	5.52	5.48
0.85	0.86	0.88	0.90	0.92	0.94	0.96	4.90	5.73	5.69	5.66	5.62	5.58	5.54
0.90	0.91	0.92	0.94	0.96	0.98	1.00	4.95	5.80	5.76	5.73	5.69	5.65	5.61
0.95	0.96	0.97	0.99	1.01	1.03	1.05	5.00	5.87	5.83	5.79	5.75	5.71	5.67
1.00	1.02	1.03	1.04	1.06	1.08	1.10	5.05	5.94	5.90	5.86	5.82	5.78	5.74
1.05	1.07	1.08	1.09	1.11	1.13	1.15	5.10	6.01	5.96	5.92	5.88	5.84	5.80
1.10	1.12	1.13	1.14	1.16	1.18	1.20	5.15	6.08	6.03	5.99	5.95	5.91	5.87
1.15	1.17	1.18	1.19	1.21	1.23	1.25	5.20	6.15	6.10	6.06	6.01	5.97	5.93
1.20	1.22	1.23	1.24	1.26	1.28	1.30	5.25	6.22	6.17	6.13	6.08	6.04	6.00
1.25	1.28	1.29	1.30	1.32	1.34	1.36	5.30	6.30	6.25	6.20	6.15	6.11	6.06
1.30	1.33	1.34	1.35	1.37	1.39	1.41	5.35	6.37	6.32	6.27	6.22	6.17	6.12
1.35	1.39	1.40	1.41	1.43	1.45	1.47	5.40	6.44	6.39	6.34	6.29	6.24	6.19
1.40	1.44	1.45	1.46	1.48	1.50	1.52	5.45	6.52	6.46	6.41	6.36	6.31	6.26
1.45	1.50	1.51	1.52	1.54	1.56	1.58	5.50	6.59	6.53	6.48	6.43	6.38	6.33
1.50	1.55	1.56	1.57	1.59	1.61	1.63	5.55	6.66	6.60	6.55	6.49	6.44	6.39
1.55	1.61	1.62	1.63	1.65	1.67	1.69	5.60	6.73	6.67	6.62	6.56	6.51	6.45
1.60	1.66	1.67	1.68	1.70	1.72	1.74	5.65	6.81	6.74	6.69	6.63	6.57	6.51
1.65	1.72	1.73	1.74	1.76	1.78	1.80	5.70	6.88	6.81	6.76	6.70	6.64	6.58
1.70	1.77	1.78	1.80	1.82	1.84	1.86	5.75	6.95	6.88	6.83	6.77	6.71	6.65
1.75	1.83	1.84	1.86	1.88	1.90	1.92	5.80	7.03	6.96	6.90	6.84	6.78	6.72
1.80	1.88	1.89	1.91	1.93	1.95	1.97	5.85	7.11	7.04	6.98	6.92	6.86	6.80
1.85	1.94	1.95	1.97	1.99	2.01	2.03	5.90	7.18	7.11	7.05	6.99	6.93	6.87
1.90	1.99	2.00	2.02	2.04	2.06	2.08	5.95	7.25	7.18	7.12	7.06	7.00	6.93
1.95	2.05	2.06	2.08	2.10	2.12	2.14	6.00	7.33	7.26	7.19	7.13	7.07	7.00
2.00	2.10	2.11	2.13	2.15	2.17	2.19	6.05	7.40	7.33	7.26	7.20	7.13	7.06
2.05	2.16	2.17	2.19	2.21	2.23	2.25	6.10	7.48	7.41	7.34	7.27	7.20	7.13
2.10	2.21	2.22	2.24	2.26	2.28	2.30	6.15	7.55	7.48	7.41	7.34	7.27	7.20
2.15	2.27	2.28	2.30	2.32	2.34	2.36	6.20	7.63	7.56	7.49	7.42	7.35	7.28
2.20	2.33	2.34	2.36	2.38	2.39	2.41	6.25	7.71	7.64	7.56	7.49	7.42	7.35
2.25	2.39	2.40	2.42	2.44	2.45	2.47	6.30	7.79	7.72	7.64	7.57	7.50	7.43
2.30	2.44	2.45	2.47	2.49	2.50	2.52	6.35	7.87	7.80	7.72	7.65	7.57	7.50
2.35	2.50	2.50	2.52	2.54	2.55	2.57	6.40	7.94	7.87	7.79	7.72	7.64	7.57
2.40	2.56	2.56	2.58	2.59	2.60	2.62	6.45	8.02	7.95	7.87	7.80	7.72	7.65
2.45	2.62	2.62	2.64	2.65	2.66	2.68	6.50	8.10	8.03	7.95	7.87	7.79	7.72
2.50	2.67	2.67	2.69	2.70	2.71	2.73	6.55	8.18	8.10	8.02	7.94	7.86	7.79
2.55	2.73	2.73	2.75	2.76	2.77	2.79	6.60	8.26	8.18	8.10	8.02	7.94	7.86
2.60	2.79	2.79	2.81	2.82	2.83	2.85	6.65	8.34	8.26	8.18	8.10	8.02	7.94
2.65	2.85	2.85	2.87	2.88	2.89	2.91	6.70	8.42	8.34	8.26	8.18	8.10	8.02
2.70	2.91	2.91	2.93	2.94	2.95	2.97	6.75	8.50	8.42	8.34	8.26	8.17	8.09
2.75	2.97	2.97	2.98	2.99	3.01	3.03	6.80	8.58	8.49	8.41	8.33	8.24	8.16
2.80	3.03	3.03	3.04	3.05	3.07	3.08	6.85	8.66	8.57	8.49	8.40	8.31	8.23
2.85	3.09	3.09	3.10	3.11	3.12	3.13	6.90	8.74	8.65	8.57	8.48	8.39	8.31
2.90	3.15	3.15	3.15	3.16	3.17	3.18	6.95	8.82	8.73	8.65	8.56	8.47	8.38
2.95	3.21	3.21	3.21	3.21	3.22	3.23	7.00	8.91	8.82	8.73	8.64	8.55	8.46
3.00	3.27	3.27	3.27	3.27	3.28	3.29	7.05	8.99	8.90	8.81	8.72	8.63	8.54
3.05	3.33	3.33	3.33	3.33	3.34	3.35	7.10	9.07	8.98	8.89	8.80	8.71	8.62
3.10	3.39	3.39	3.39	3.39	3.40	3.41	7.15	9.15	9.06	8.97	8.88	8.79	8.70
3.15	3.45	3.45	3.45	3.45	3.46	3.47	7.20	9.23	9.14	9.05	8.96	8.87	8.78
3.20	3.51	3.50	3.50	3.50	3.51	3.52	7.25	9.31	9.22	9.13	9.04	8.95	8.86
3.25	3.57	3.56	3.56	3.56	3.57	3.58	7.30	9.40	9.31	9.22	9.12	9.03	8.94
3.30	3.63	3.62	3.62	3.62	3.63	3.64	7.35	9.48	9.39	9.30	9.20	9.11	9.01
3.35	3.69	3.68	3.68	3.68	3.69	3.69	7.40	9.57	9.48	9.38	9.28	9.19	9.09
3.40	3.75	3.74	3.74	3.74	3.74	3.74	7.45	9.65	9.56	9.46	9.36	9.27	9.17
3.45	3.81	3.80	3.80	3.80	3.80	3.80	7.50	9.74	9.65	9.55	9.45	9.36	9.26
3.50	3.87	3.86	3.86	3.86	3.86	3.86	7.55	9.82	9.73	9.63	9.53	9.44	9.34
3.55	3.94	3.93	3.93	3.92	3.92	3.92	7.60	9.91	9.81	9.71	9.61	9.52	9.42
3.60	4.00	3.99	3.99	3.98	3.98	3.98	7.65	10.00	9.90	9.80	9.70	9.61	9.51
3.65	4.06	4.05	4.05	4.04	4.04	4.04	7.70	10.09	9.99	9.89	9.79	9.70	9.60
3.70	4.13	4.12	4.12	4.11	4.10	4.10	7.75	10.18	10.08	9.98	9.88	9.78	9.68
3.75	4.20	4.19	4.18	4.17	4.16	4.16	7.80	10.26	10.16	10.06	9.96	9.86	9.76
3.80	4.26	4.25	4.24	4.23	4.22	4.22	7.85	10.35	10.25	10.15	10.05	9.95	9.84
3.85	4.32	4.31	4.30	4.29	4.28	4.27	7.90	10.44	10.34	10.24	10.14	10.03	9.92
3.90	4.39	4.37	4.36	4.35	4.34	4.33	7.95	10.52	10.42	10.32	10.22	10.11	10.00
							8.00	10.61	10.51	10.40	10.30	10.19	10.09

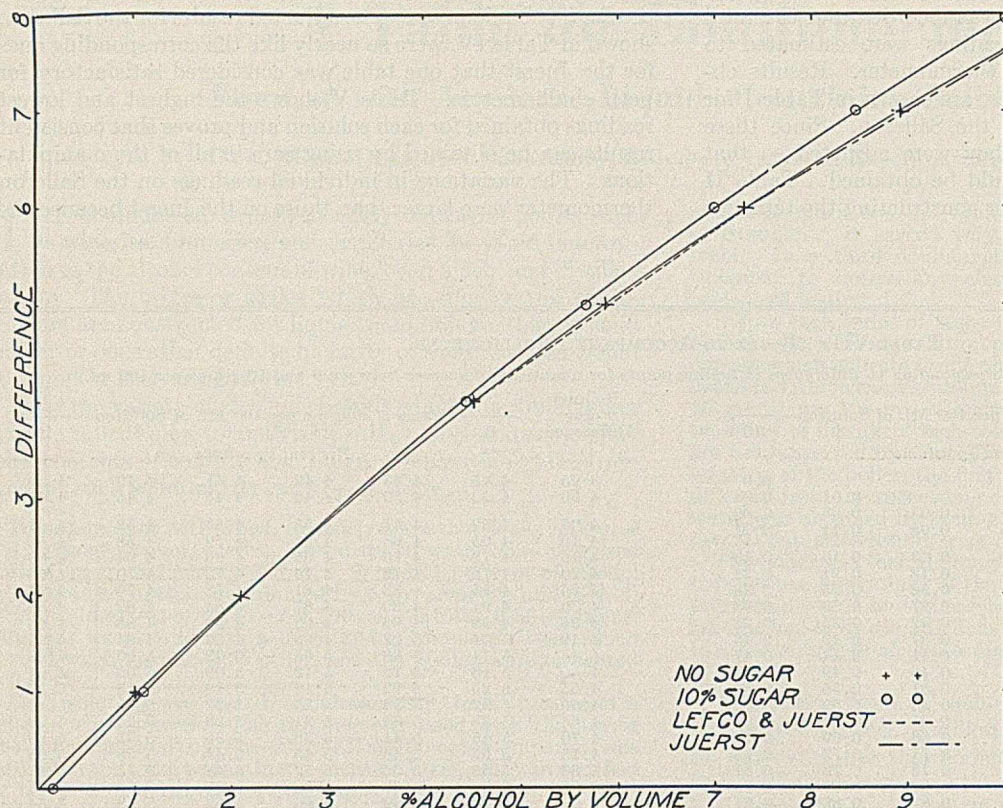


FIGURE 1

scale divisions of the Salleron are smaller and consequently more difficult to read.

A formula was obtained for each series of solutions containing the same quantity of sucrose, by use of the equation $y = ax + bx^2 + cx^3$, in which y is the difference between boiling points, x is the per cent of alcohol by volume, and a , b , and c are constants whose values were calculated from the experimental data, by the method of least squares. These formulas are shown in Table VI. By their use Table VII was constructed, showing per cent of alcohol by volume, for each 0.05 degree difference between boiling points, from 0 to 8, for solutions containing 0, 2, 4, 6, 8, and 10 grams of sucrose per 100 ml. This table may be used with any ebulliometer whose thermometer is based on the Centigrade scale. Using boiling point differences as ordinates and percentage of alcohol as abscissas, the author plotted a curve for each quantity of sucrose used. These curves are shown in Figure 1.

The column of Table VII for solutions containing no sucrose agrees very well with the table which accompanies the Lefco ebulliometer (3) except at one point. When the boiling point differences in the Lefco table are plotted against percentages of alcohol, they form a smooth curve, except for the boiling point differences 4.00 to 6.00, where they form a straight line. Evidently the interpolations at that part of the table were made by proportion rather than by use of a formula. Almost exactly the same discrepancy is noticed at the corresponding part of the Juerst table (2).

At boiling point difference 6.20, Table VII and the Juerst table again begin to separate and at 7.75, the highest point on the Juerst scale, the discrepancy is 0.07 per cent alcohol. Repeated attempts to check the Juerst table at its upper limit were unsuccessful. Table VII agrees very well with the Lefco table at its upper limit. It is concluded that there is a small error in the Lefco table at boiling point difference 5.00 and in

the Juerst table at boiling point difference 5.00 and from 6.20 to 7.75.

A correction table (1) to be used for beer and beverages having a real extract (solids) of from 3 to 12 per cent gives a factor to be added to the alcohol found, according to the amount of solids present. It agrees exactly with Table VII at 5.66 per cent alcohol, for the various percentages of sucrose, but it cannot be used for liquids containing other percentages of alcohol.

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- (2) U. S. Internal Revenue Regulations, 7, Table VII.
- (3) *Ibid.*, Table VIII.

A Modified Jones Reductor

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THE Jones reductor is a convenient apparatus for effecting the reduction of certain compounds. Its efficiency in certain cases, however, is governed by the rate at which the solution containing the substance to be reduced flows through the reductor. A definite rate of flow through a Jones reductor equipped with an ordinary stopcock can be obtained only by trial and error; furthermore, duplicate rates are practically impossible.

The modified Jones reductor herein described enables one to obtain a definite and constant rate of flow. This may be accomplished by fitting the delivery tube of the reductor with replaceable ground-glass capillary tubes of various-sized orifices.

The tip of the delivery tube is first expanded to form the female joint. A 15-cm. (6-inch) length of capillary tubing, possessing an inside diameter of 1.5 mm. and an outside diameter approximately equal to that of the delivery tube of the reductor, is heated at one end until the orifice is barely closed. The capillary tube is then heated at a point 5 cm. (2 inches) from the closed end and drawn out until it possesses a taper simulating that of the female cone. The male cone is obtained by cutting the tubing at the constriction. Three or four male cones should be made and ground into the delivery tube of the reductor, care being taken not to grind too long on any one cone before grinding in the next. In this way all the cones are gradually ground to the same size and may be used interchangeably in the reductor. The closed tips of the cones are then ground down on a piece of plate glass until each cone possesses the desired orifice size. In this way cones delivering approximately 20, 50, 100, or any desired number of milliliters per minute may be obtained. The volume delivered must be determined by trial. If too large a volume is delivered, the orifice may be closed slightly by reheating the tip.

Device for Subliming Iodine

JACOB CORNOG AND LEONARD OLSON

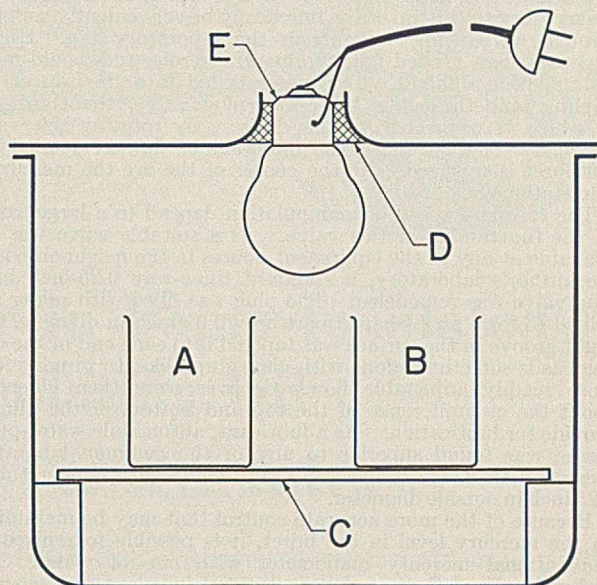
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MANY books on analytical chemistry give directions for subliming iodine in quantities of a few grams, but the authors have not found in the literature a description of a device that may be assembled from materials found in most laboratories and used for subliming iodine in lots ranging up to 500 grams (1 pound) in weight. Such a device is described in this article.

As indicated in the figure, the device consists of a desiccator of Pyrex glass, having a diameter of approximately 250 mm., and bearing a 40- to 60-watt light bulb in the lid. Lead wires from an ordinary municipal lighting circuit are soldered to the metallic base, *E*, of the bulb, which is sealed in the hole in the desiccator lid with plaster of Paris, *D*, in such a way that no metallic surface is exposed to the interior of the desiccator and also so that the plaster does not touch the wire soldered to the central terminal of the bulb, which is covered with de Khotinsky or a similar cement. The tile, *C*, supports the beaker or evaporating dish, *A*, containing the iodine to be sublimed and the beaker, *B*, containing a drying agent such as phosphorus pentoxide. If a resistance is placed in series in the external circuit, closer regulation of heating temperatures is assured.

When a current is passed through the bulb, the top of the desiccator becomes warmer than the bottom and iodine vaporizes and condenses on the lower sides and the bottom. The rate of sublimation may be increased by using devices which increase the difference in temperature existing between the upper and lower parts of the desiccator, such as insulating the top or cooling the bottom of the desiccator.

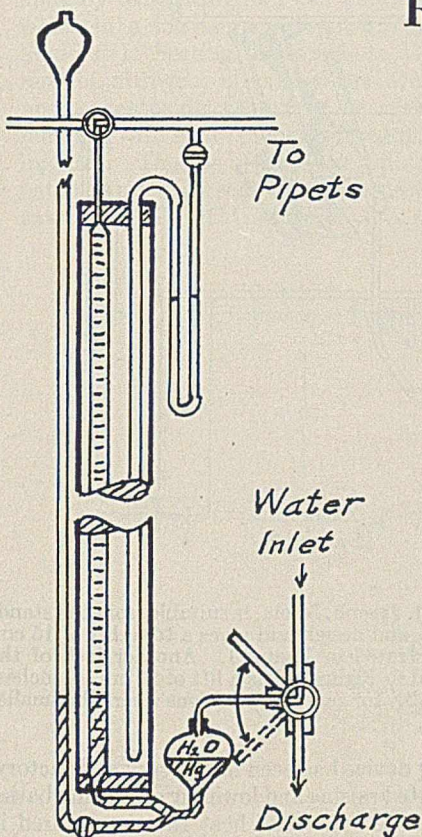
This apparatus has found a variety of uses in this laboratory, such as drying and subliming iodine recovered from accumulated iodine "residues" by wet methods, recovering



iodine mixed with broken glass and dirt resulting from the breaking of bottles in transit or by dropping them on the floor, and in the drying and purification of substances, such as iodine monochloride, that are volatile, corrosive, and easily decomposed at relatively low temperatures.

Rapid Operating Device for Orsat Apparatus

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THE operation of ordinary gas analysis equipment is somewhat tedious and requires the constant attention of the analyst. Automatic absorption pipets (1, 2, 3) and a hydraulically operated device for transporting the mercury bulb (4) have been suggested to decrease the time consumed and to relieve the operator during a portion of the analysis. Each has a advantageous application.

A study of the operation of a standard analysis procedure will indicate that a large portion of the total time is used in returning pipet solutions to their original marks, balancing the pressure, and reading the buret. These operations are complicated by the fact that the confining liquid in the buret is not perfectly controlled, but is free to surge between the buret and the leveling bulb, and cannot be brought to rest quickly.

The device described below permits a positive control on the confining liquid (mercury must be used) by the presence of an incompressible fluid (water) over the mercury in the leveling bulb. This means of control so facilitates the operation of any type of gas analysis equipment that considerable saving of time should result. In the analysis of coal-mine air (carbon dioxide, oxygen, carbon monoxide, methane) with a standard laboratory-type apparatus, using contact and slow-combustion pipets, the total time of analysis may be cut in half with this device.

Description

The essential difference between this device and the conventional Orsat apparatus is that the motivating force here is water pressure which is applied over the mercury in the leveling bulb. This change necessitates the use of a leveling tube (a tube parallel to the buret, open at the top and connected at the bottom to the buret and to the leveling bulb) so that an estimate of the pressure in the buret may be obtained if desired. The usual long rubber

tube connecting the leveling bulb to the buret is replaced by short lengths of glass tubing connecting the leveling bulb to the buret and to the leveling tube. A stopcock is inserted between the leveling bulb and the leveling tube.

Water at a pressure slightly in excess of that required to raise the mercury to the top of the buret may be conveniently supplied through a reducing valve from the laboratory tap. Higher pressures than needed might prove dangerous and would make manipulation difficult. The water is led into the top of the leveling bulb through a three-way plug valve, the third side of which is connected to the drain. By rotating this valve through a 90-degree arc the direction of flow of mercury in the buret is reversed. At the center of the arc the mercury is held stationary.

The ease and speed of manipulation depend to a large extent on the functioning of this valve. As a suitable valve was not available at any of the equipment houses in the neighborhood of the author's laboratory, a standard three-way 0.25-inch brass plug valve was remodeled. The plug was filled with solder and drilled to form an L-shaped port only 0.09 inch in diameter. A slight groove in the surface was tapered into each end of the drill hole, as is sometimes done with glass stopcocks, to provide for a more readily adjustable flow. Grooves were then machined about the circumference of the top and bottom of the plug to provide for lubrication. As a lubricant, automobile water-pump grease was found superior to any of the common laboratory greases. All water connections were made with copper tubing 0.25 inch in outside diameter.

Because of the more accurate control that may be maintained on the mercury level in the buret, it is possible to replace the conventional mercury manometer with one of water. This makes leveling off somewhat easier and increases the accuracy of the apparatus. The flow of water over the mercury in the leveling bulb serves to keep the mercury clean, and it has not

been necessary throughout the analysis of over a thousand samples of mine air to provide any additional cleaning.

Operation

Operation is similar to the conventional procedure except that the mercury is raised in the buret by passing water into the top of the leveling bulb instead of raising the leveling bulb. In leveling off, the mercury level in the leveling tube is adjusted to approximately that in the buret. Then the stopcock is closed to the leveling tube, the stopcock to the water manometer opened, and the pressure balanced with that in a conventional compensating tube.

After the operator is familiar with the apparatus, an estimate of pressure may be obtained from returning the solutions in the absorption pipets to their original marks and the use of the leveling tube may be largely eliminated, affording an additional saving of time.

This equipment, in addition to saving time, takes much of the drudgery out of routine analysis by eliminating the continual raising and lowering of the mercury-filled leveling bulb. It is particularly well suited to procedures that require a slow or even flow of gas, such as slow combustion.

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- (3) Egerton, A. C., and Pidgeon, L. M., *J. Sci. Instruments*, 8, 234 (1931).
- (4) Tauch, E. J., *IND. ENG. CHEM.*, 19, 1349 (1927).

A Laboratory Lifting Device

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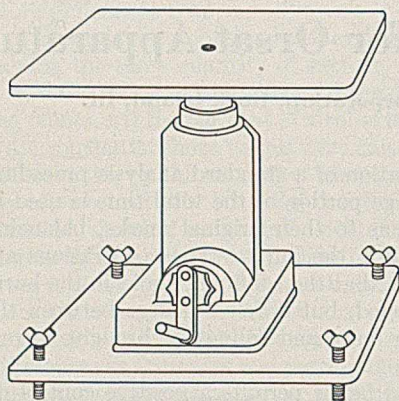


FIGURE 1

MANY operations in the laboratory call for the lowering and raising of a heating bath, such as an oil bath under a reaction flask or a still. This operation is normally accompanied by a decided hazard on account of the difficulty of handling a container of hot oil, especially when it must be

done quickly to prevent a reaction from getting out of control. A very simple device for doing this, which has been used in this laboratory for several years, is illustrated in Figure 1.

It consists of an automobile jack mounted on a plate equipped with leveling screws which are long enough to enable the plate to clear the base of a ring stand. The jack is equipped on top with a plate approximately 20 × 20 cm. which is made to support the oil bath, with or without a hot plate, or any other piece of equipment. Several makes and designs of automobile jacks have been tried out. The hydraulic type is unsatisfactory because it will not come down readily except with a large load, and also because raising and lowering require two different kinds of lever operation. The ratchet type is also unsatisfactory because an up-and-down lever operation might result in either raising or lowering, depending on the setting of the trip control lever.

The preferred type is that calling for a rotary motion clockwise for raising and counterclockwise for lowering. The dreadnaught jack No. 26, 1-ton capacity, made by the Auto Specialties Manu-

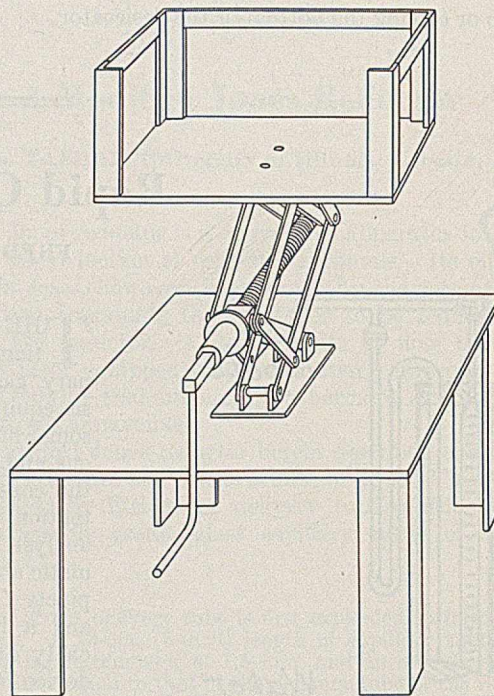
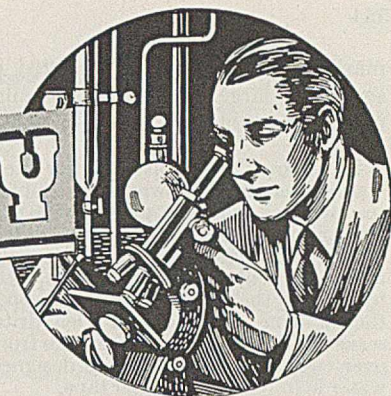


FIGURE 2

facturing Company, St. Joseph, Mich., is suitable from the standpoint of size, stability, and design and gives a total lift of 15 cm. (6 inches). It is illustrated in Figure 1. Another jack of the scissor type, illustrated in Figure 2, has a lift of 23 cm. (9 inches) but is unnecessarily bulky for general operations where the smaller jack has sufficient lift.

This type of lifting device has been found very satisfactory, not only for the repeated raising and lowering of heating baths, but also for the accurate control of heat that is required in fractional distillation.

MICROCHEMISTRY



Determination of Water in Paper-Insulated Cables and Insulating Oil

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THE determination of water in insulating oil, and more especially oil-impregnated paper of high-voltage cable, has received considerable attention by analysts because of its deleterious effect when present in insulating material. The procedures which have been used in the past may be divided into two main classes: gas evolution and water evolution. The former group comprises reagents such as sodium, yielding hydrogen (7); calcium carbide, yielding acetylene (1); sodamide, giving ammonia; and Grignard reagents—for example, methyl magnesium halide—producing methane (8).

In the second group the water is removed in such a manner that it is possible to ascertain its weight or volume. This is done by evacuating at room temperature and retaining the water in a low-temperature trap (solid carbon dioxide or liquid air); heating the sample to 110° to 150° C. in a current of nitrogen, absorbing the evolved material in phosphorus pentoxide, Dehydrite, or calcium chloride (2, 13, 14); refluxing with high-boiling hydrocarbon (3, 16), and centrifuging (15). The use of the quartz fiber balance for unimpregnated paper has been employed successfully by some investigators (11, 17). Critical solution in aniline (19) and heat of

hydration (10) have been used, together with many modifications of the above procedures (6). Electrical methods are inherently unsatisfactory for the determination of water.

The general criticism which may be directed at the first group of procedures is that the reagent combines with other constituents in the sample. The Grignard reagent, for example, may evolve methane by reaction with alcohols, acids, peroxides, and some hydrocarbons and, until a correction be applied to the result, the percentage of water so obtained is in error. A correction is obtained with some uncertainty for acids (5) but the other interfering substances are extremely difficult to determine in small amounts. Removal of the water by heat or evacuation, as called for in the second procedure, is almost impossible to accomplish without at the same time carrying along an impurity which would cause one to doubt the actual weight of water obtained by absorption. When the sample contains cellulose, the problem of total elimination of water presents itself, making it necessary to decide between water of absorption (free, 18), and water of composition (bound). The manner in which some of the difficulties of the second procedure are treated experimentally is described in the present paper. The apparatus is pri-

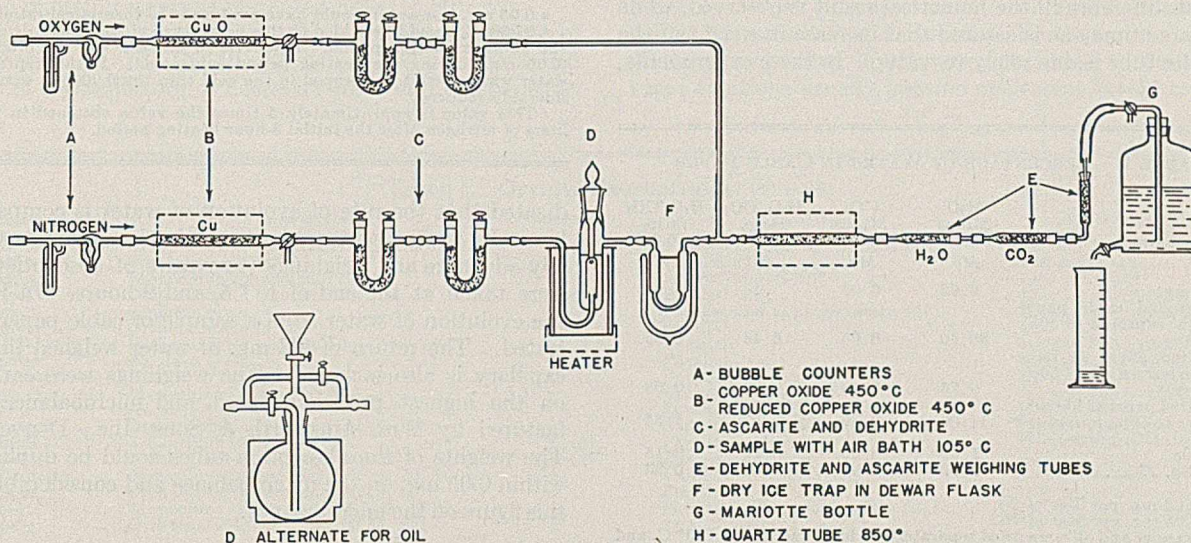


FIGURE 1. DIAGRAM OF APPARATUS FOR THE DETERMINATION OF WATER

marily applicable to impregnated paper tape, but where a somewhat larger oil sample is available a slight modification makes it suitable for analysis of water in oil.

In Figure 1, the apparatus for the determination of water is diagrammatically shown. The bubble counters, *A*, enable one to observe a constant gas velocity in the separate gas streams. The actual volume of gas is measured at the exit end of the train by the Mariotte bottle, *G*. This value determines the time of the experiment. The purification of the nitrogen is accomplished by passing the gas over reduced copper oxide at 450° C., *B*, and thence over Ascarite and Dehydrite, *C*. The same furnace serves to remove the combustibles from the oxygen in its passage over copper oxide. Cell *D* is designed for the determination of water in impregnated paper tape.

The perforated cylinder sealed to the ground-glass stopper serves as a receptacle for the sample. A minimum of handling of the sample is accomplished by merely poking the paper tape into the cylinder, cutting the ends which were handled, weighing, and immersing in the heated oil as the stopper is put in place. The oil in which the sample is immersed is the type that is used in diffusion pumps such as *n*-butyl phthalate or Apiezon oil. The latter is practically nonvolatile and is maintained at a temperature of 105° to 110° C. The quartz tube at *H* is filled with quartz fragments and held at a temperature of 800° to 850° C. The microchemical absorption tubes at *E* were prepared, weighed, and handled as discussed by Pregl (12) and Niederl (9). The cold trap, *F*, retains the expelled water and other condensable constituents but allows the passage of any hydrogen, methane, carbon monoxide, and other carbonaceous substances which if present would render the carbon dioxide correction extremely uncertain.

The general scheme of analysis is apparent from the description of the apparatus. The novel points are the immersion of the sample in hot oil, thus ensuring good thermal contact with rapid evolution of water, and the application of a correction for the volatile hydrocarbon constituents obtained by burning them and calculating the water correction from the carbon dioxide weight (4). The general formula C_nH_{2n} is arbitrarily chosen as representing the evolved carbonaceous material and thus for every milligram of carbon dioxide found, 0.4 mg. is subtracted from the weight of the evolved water. This correction was generally not large—0.5 mg. of water or less—and should represent not more than the equivalent of 5 to 10 per cent of the total water evolved. When comparatively large amounts of carbon dioxide are encountered, the method suffers in accuracy, but it is evident that the result would be more uncertain when the possibility of the absorption of hydrocarbon by the Dehydrite is considered if the expedient were not taken. In Table I, the experimental results are given in which the percentage of water for a 3-liter gas flow is listed as calculated from a ratio of 0.4 and 0.6. No great difference in the numerical result is observed, while at the same time one is assured that increase in weight of the Dehydrite tube is due solely to water. In these experiments,

TABLE I. PERCENTAGE OF WATER IN CABLE PAPER

Sample	H ₂ O Minus Blank ^a Mg.	CO ₂ Minus Blank ^b Mg.	H ₂ O	
			H ₂ O/CO ₂ ratio, 0.4	H ₂ O/CO ₂ ratio, 0.6
			%	%
H ₂ O, 9.8 mg.	9.95	0.00
Unimpregnated cable paper (30 days' exposure to 50% humidity)	30.40	0.05	6.46	6.46
Impregnated paper (5 min- utes exposure to 50% humidity) ^c	0.75	0.40	0.09	0.08
Impregnated paper (24 hours exposure to 50% humidity) ^c	11.00	0.30	1.57	1.55
1936—solid, CO ₂ -purged, 27- kv. cable	1.50	0.50	0.17	0.15
1923—solid, 27-kv. cable	2.70	0.90	0.36	0.33

^a Av. 0.03 mg. per liter of N₂.

^b Av. 0.05 mg. per liter of N₂.

^c Cable paper and oil were dried separately under a vacuum at 100° C. and the paper was impregnated before breaking vacuum.

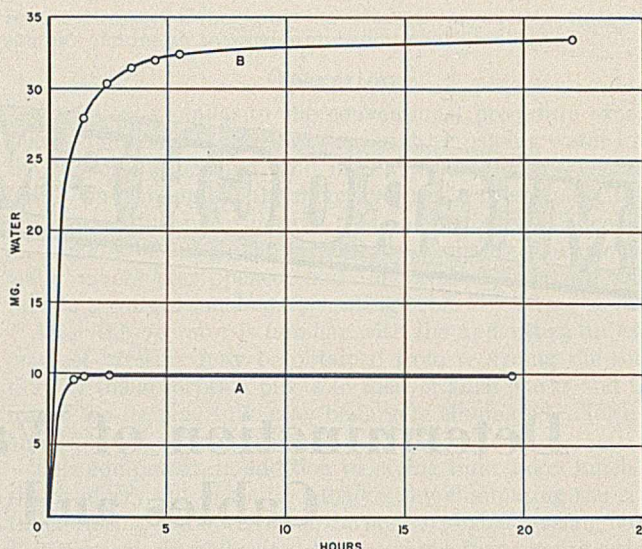


FIGURE 2. RATE OF REMOVAL OF WATER

A. 9.8 mg. of water

B. 470 mg. of unimpregnated cable paper previously exposed to air of 50 per cent relative humidity for 1 month

it was previously established that no difference in the experimental results was obtained whether or not the cold trap was used.

One of the chief drawbacks with all methods for the determination of water in cellulose products is the difficulty of removal of the last trace of water from the sample. If one wishes to avoid an arbitrary time limit to distinguish between free and bound water, it is possible to follow the time-water evolved curve and stop the experiment when it is in-

TABLE II. DETERMINATION OF WATER IN PYRANOL

	Nitrogen Liters	H ₂ O Mg	CO ₂ Mg.	Ratio
Blank	1	0.06	0.02	...
Trap in place	3
Trap removed	2	11.19	7.74	1.45
1 additional		0.47	3.52	0.116 ^a
1 additional		0.52	3.30	0.130
		Gross	12.18	14.56
		Less blank	11.94	14.48
		CO ₂ correction	1.48	...
		Net water	10.46	...
		Water, % ^b	0.0035	...
Trap in place	15
Trap removed	2	5.86	38.04 ^c	0.154
1 additional		2.53	18.18	0.140

^a 0.05 mg. less water would have produced the theoretical ratio.

^b 0.0008% should be added to this percentage of water as calculated from the 15-hour heating period. However, in no case was the original blank (0.06 mg.) during the heating period subtracted. If this represents free water which would be trapped in the cold trap the 0.0008% would be considerably reduced.

^c This value is approximately 5 times the value obtained in the first 2 liters of nitrogen after the initial 3-hour heating period.

indicated that the rate of evolution of water is comparatively low. In the present apparatus, 2-hour immersion was usually adequate and weighings (two pairs of absorption tubes) were taken at the end of 1, 1.5, and 2 hours. In Figure 2, the evolution of water from a sample of cable paper is illustrated. The return of 9.8 mg. of water weighed in a small capillary is also included. The weighings were carried out on the highest precision macro- and microbalances manufactured by Wm. Ainsworth & Sons, Inc., Denver, Colo. The weights of the absorption tubes could be duplicated to within 0.05 mg. on the macrobalance and considerably below this figure on the microbalance.

Where large samples (300 grams) of insulating oil are available, the alternate cell (*D*, Figure 1) may be used. Several samples of

Pyranol—a nonflammable transformer oil—were analyzed for water content, the results ranging from 0.002 to 0.004 per cent of water by weight. In these experiments the cold trap, solid carbon dioxide and methanol, was employed. The Pyranol, maintained at a temperature of 105° C. for 3 hours, was freed of water by passage of the purified nitrogen. The water and Pyranol vapors were retained by the cold trap. The trap was now removed, the Pyranol cell was isolated by means of the by-pass, and the water and Pyranol vapors were carried into the combustion furnace where combustion took place. The halogen was retained in the furnace by means of silver wool and from the ratio of water to carbon dioxide established on dry Pyranol, the net water may be readily computed.

The ratio $C_{2n}H_nCl_n$ (aromatic) is desired by the manufacturer in order that the hydrogen and chlorine may be present in equivalent amounts. The ratio H_n/C_{2n} in terms of water and carbon dioxide equals 0.102. This value was never reached in actual experiment, as is shown in Table II.

Advantages of Method

The procedure enables one to determine water per se without the possibility of contamination of the absorbent.

The net water may be calculated by means of a correction based on the ratio of water to carbon dioxide.

The extent of the removal of water from the sample may be judged by periodic weighings.

The determination of water is as rapid as is commensurable with accuracy.

Acknowledgment

The authors wish to express their appreciation of the interest taken in this work by W. F. Davidson, Director of Research.

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Microscopic Identification of Sugars

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THIS study was undertaken because there is no exclusively characteristic test for glucose and some other sugars (?). Refractive indices and certain other optical properties have been used to identify sugars (22, 42, 43), but the determination of these constants is time-consuming. The crystal habit of a few sugars has also been used for purposes of identification (8, 24, 26, 32) and it is desirable to extend this use of the crystal habit to the whole group. Microscopic fields containing sugar crystals can easily be compared for identity with the photomicrographs presented here ($\times 80$).

Since the habit of a crystal is affected by the conditions of its formation (9, 12, 17, 18, 20, 21, 33, 39), a prescribed method of crystallization is necessary for useful comparisons.

Wernicke (41) and Hudson and Yanovsky (19) have successfully crystallized sugars from water solutions by addition of acetic acid or alcohol. This paper recites the use of alcohol, acetone, acetonitrile, and 1,4-dioxane in obtaining photomicrographs showing a distinctive crystal habit for each of eighteen sugars in one or more of these solvents.

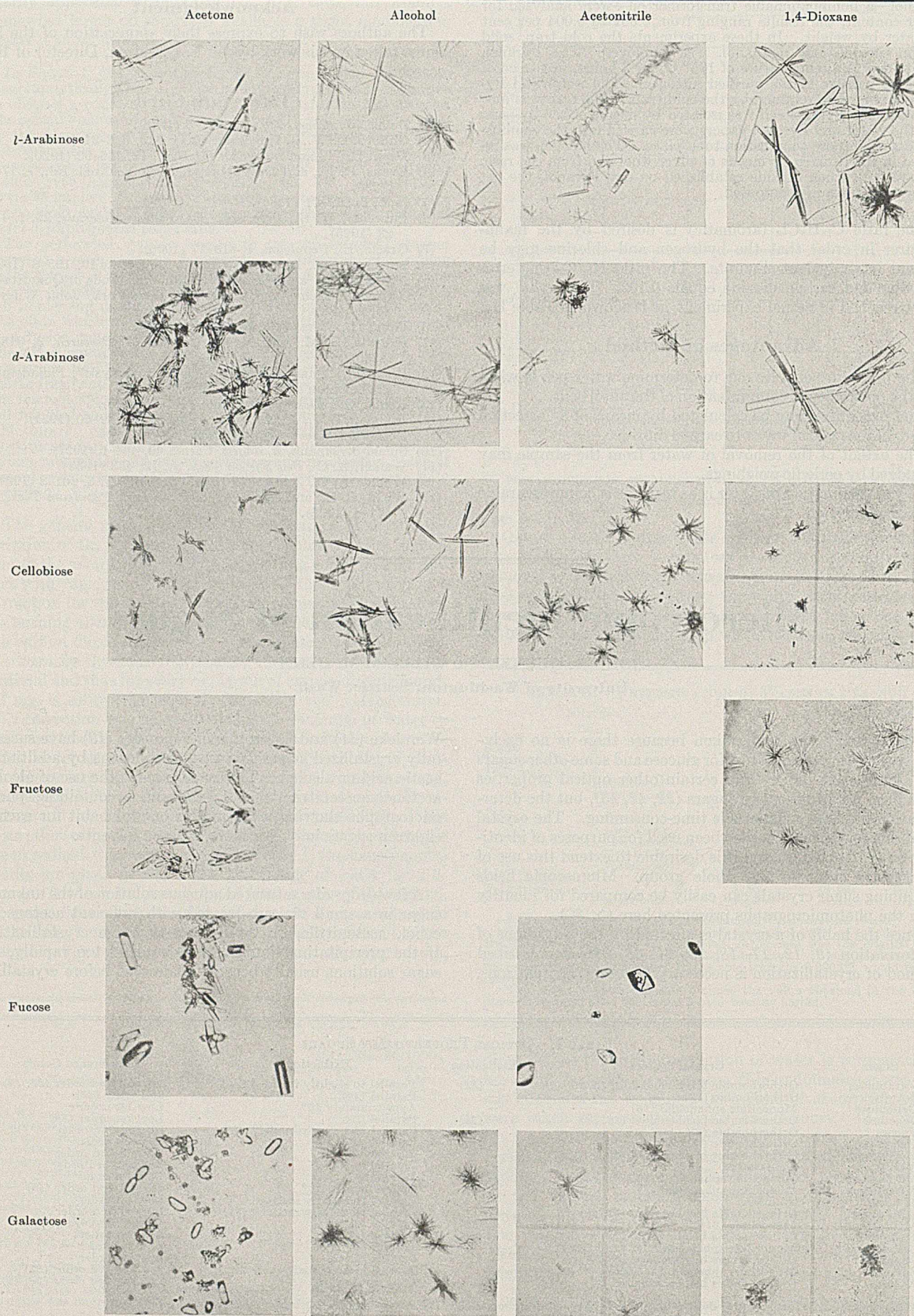
Method

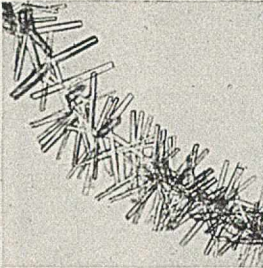
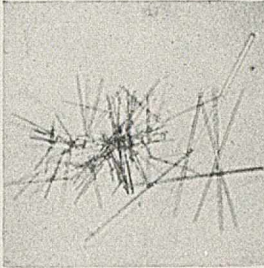
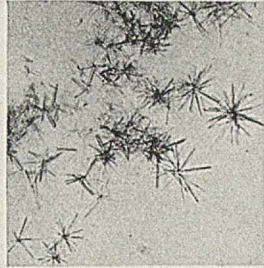


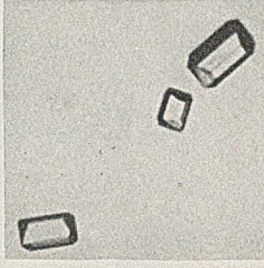
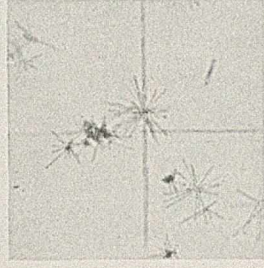
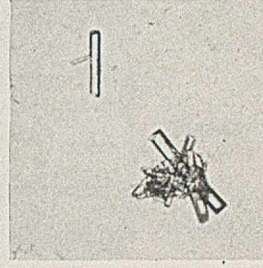

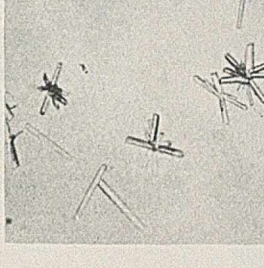
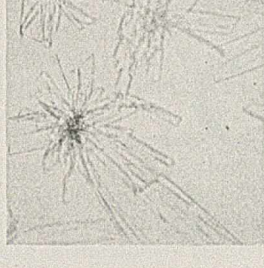






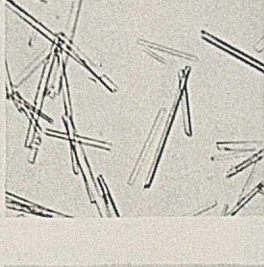
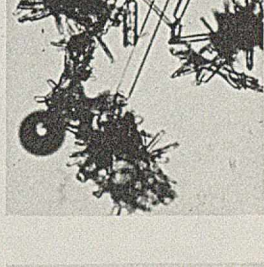

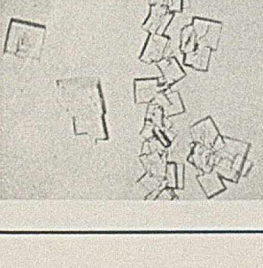
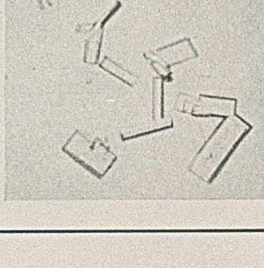
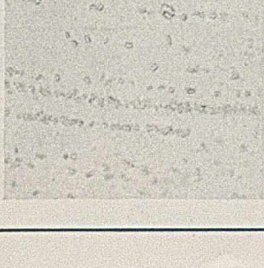
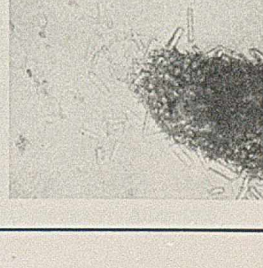
A few drops of a saturated aqueous solution of the unknown sugar in a small vial are treated with sufficient acetone, alcohol, acetonitrile, or 1,4-dioxane to cause crystallization. If the precipitating liquids are not added too rapidly, the sugar solutions usually become opalescent before crystalliza-

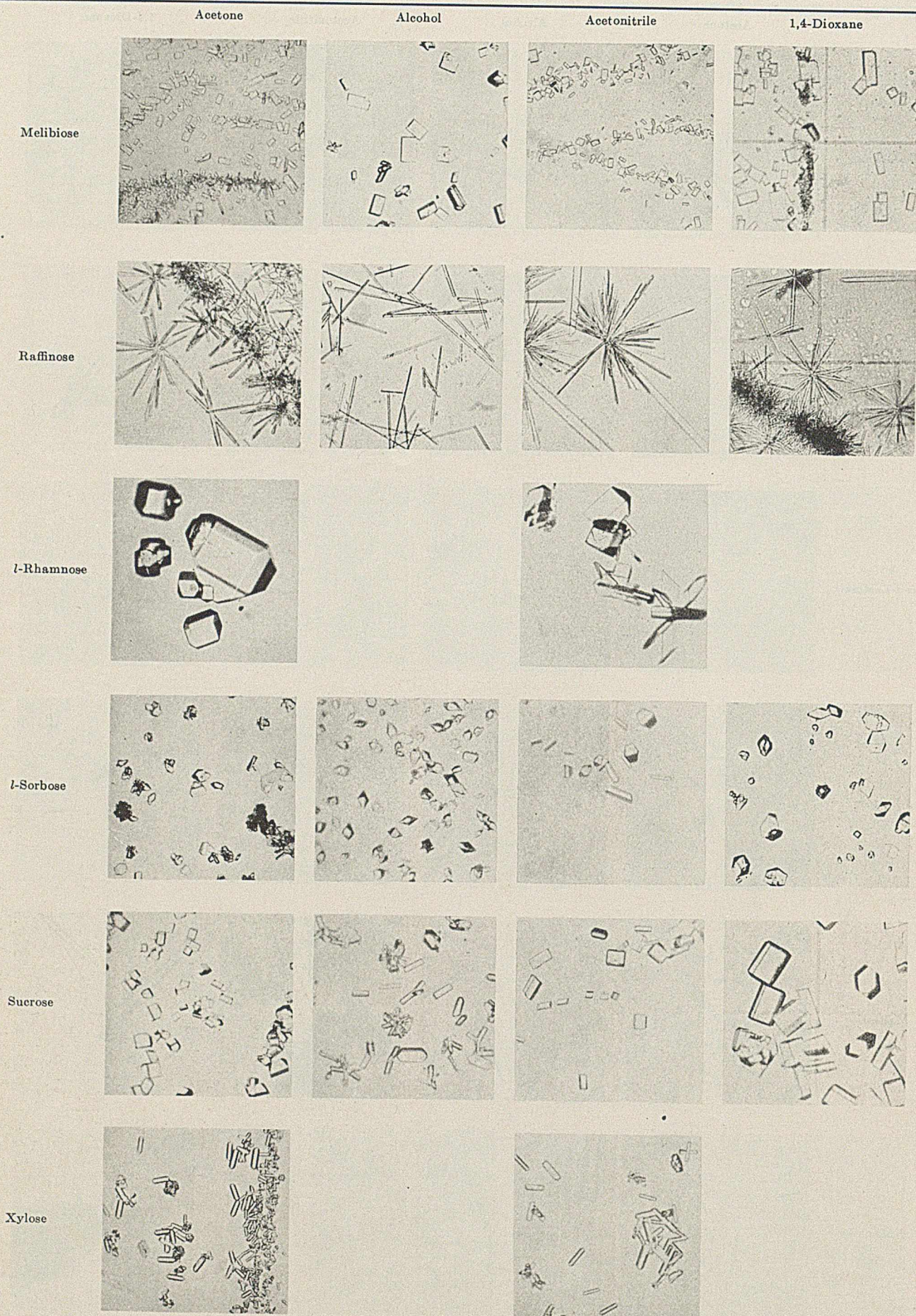
TABLE I. OPTICAL PROPERTIES OF SUGARS

Sugar	Crystal System	Elongation	Extinction	Interference Colors
<i>l</i> -Arabinose	Orthorhombic bisphenoidal (5, 14, 30, 42)	— (22)	Parallel to slightly inclined (42)	1st and 2nd order (22, 42)
<i>d</i> -Arabinose	Orthorhombic (37)	—	Parallel (42)	2nd order (42)
Cellobiose	Monoclinic sphenoidal (16)	—	Approximately 16°	Low 1st order
Fructose	Orthorhombic bisphenoidal (16, 34)	— (22)	Parallel (22)	Low 1st order (22)
Fucose	Orthorhombic (6)	—	Parallel and approximately 25°	1st and 2nd order
Galactose	Orthorhombic (6, 23)	—	Parallel	Low 1st order
α - <i>d</i> -Glucose	Orthorhombic bisphenoidal (2, 4, 16, 35)	—	Parallel	1st and 2nd order
β -Glucose	No data (35)	—	Parallel	2nd and 3rd order
α - <i>d</i> -Lactose	Monoclinic sphenoidal (36, 38)	—	Parallel	Low 1st order
β -Lactose	Monoclinic sphenoidal (43)	—	Approximately 10°	1st and 2nd order (43)
Maltose	No data	—	Parallel to slightly inclined	Low 1st order
<i>d</i> -Mannose	Orthorhombic bisphenoidal (27, 28)	±	Parallel to slightly inclined (22)	1st and 2nd order
Melibiose	Monoclinic (51)	+ (22)	Parallel (22)	1st and 2nd order
Raffinose	Orthorhombic bisphenoidal (13)	— (22)	Parallel (22)	Low 1st order
<i>l</i> -Rhamnose	Monoclinic sphenoidal (10, 11, 30, 36)	+	Parallel	Low 1st order
<i>l</i> -Sorbose	Orthorhombic bisphenoidal (3, 25)	+	Parallel	Low 1st order
Sucrose	Monoclinic sphenoidal (15, 44)	+	Parallel and approximately 22.5°	2nd and 3rd order (22)
Xylose ^a	Orthorhombic bisphenoidal (5, 30)	+	Approximately 6° (42)	2nd and 3rd order (22, 42)

^a Wherry (42) and Pionchon (29) designate xylose as monoclinic sphenoidal. The x-ray data (5, 30) were considered more reliable (1).



	Acetone	Alcohol	Acetonitrile	1,4-Dioxane
<i>α</i> -D-Glucose				
<i>β</i> -Glucose				
<i>α</i> -D-Lactose				
<i>β</i> -Lactose				
Maltose				
<i>d</i> -Mannose				



tion (17, 40). When these opalescent liquids are observed under the microscope, the crystals can be studied during growth. Varying speeds of crystallization and the formation of colloidal suspensions may render such observation impossible. If immediate crystallization does not occur, insufficient sugar has been used and another trial may be necessary. Should a sirup precipitate, crystallization can frequently be effected by scratching the slide (8). Comparison of a representative field with the photomicrographs leads to positive identification of the sugar. (This method has been used by graduate students on single sugars and binary mixtures with complete success.) Further confirmation can be made by study of the optical properties in Table I.



A
B
SUGAR MIXTURES PRECIPITATED WITH ACETONE

A. Raffinose-d-Lactose
B. Galactose-Cellobiose

The feasibility of identifying sugars in mixtures is demonstrated in the photomicrographs of raffinose and lactose and of galactose and cellobiose. Further study of mixtures will be made.

Remarks

In the application of this method, certain impure commercial samples failed to crystallize under any circumstances because, in the transition from the dissolved to the crystalline sugar, the impurities stabilized the colloidal stage.

Bacteriological culture slides are convenient for observations; however, the vials themselves are usually adequate. In general, acetone is the most useful precipitating liquid.

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A Distillation Capillary

ALEXANDER O. GETTLER

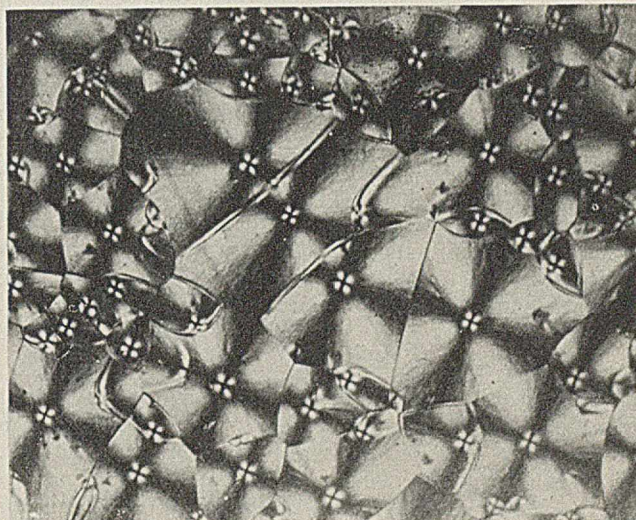
Washington Square College, New York University,
New York, N. Y.

IN USING the distillation capillary described by Gettler and Fine (1) for the fractional distillation of 0.02 to 0.06 ml. of liquid, it was found that occasionally, as in macrodistillations, a sudden violent ebullition throws the contents of the distilling bulb up into the stem of the apparatus. This tendency has been overcome by introducing asbestos fibers into the bulb of the apparatus.

The distillation capillary is made as described by Gettler and Fine; but before attempting to form the bulb, a small quantity of previously ignited and then cooled asbestos fibers is loosely inserted into the larger capillary to a distance of about 1 cm. from the end. The bulb is then made as described by Gettler and Fine. When the distillation capillary is completed, the asbestos in the stem is forced into the bulb by simple tapping.

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Courtesy, Mary L. Willard

LIQUID CRYSTALS OF CHOLESTERYL ACETATE

Preparation of Immersion Liquids

For the Range $n_D = 1.411$ to 1.785

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ONE of the most important techniques for the rapid identification of nonopaque minerals is the determination of their index of refraction. This is done by immersing fragments of the unknown mineral in a liquid of known index of refraction and comparing the indices of the two by the standard techniques—central and oblique illumination tests with a petrographic microscope. The immersion technique is becoming increasingly familiar to chemists because an unknown nonopaque substance, inorganic or organic, may be uniquely identified in a fraction of the time necessary for a quantitative analysis.

The methods used are described in many standard texts—e. g., Winchell (13), Rogers and Kerr (11), and Chamot and Mason (5). The chemist will be especially interested in the last-named, since these men are leaders in the application of optical methods to chemistry. Tables of indices of refraction and other optical constants of nonopaque minerals are available (9, 14). Tables of inorganic artificial substances have been published by Winchell (15), who is preparing tables for organic substances.

The desired properties of the liquids used in a set of immersion liquids have been described by Larsen and Berman (9); the properties of ideal immersion liquids have been given by M. J. Buerger (2), who states that the index of refraction, temperature coefficient, and dispersion should be a linear function of composition. The liquids described in this paper represent an attempt to combine all these properties so far as is practical. These liquids are not in general suitable for work with organic crystals, since they are solvents for most organic compounds. [For these compounds a water solution of K_2HgI_4 (Thoulet solution) has been suggested (see also 5, p. 375).]

The writers hope that the data on properties and the discussion of technique will be of use to those interested in preparing similar sets.

Winchell's tables (15) for artificial substances list 34 pages of index data; of these, only one page consists of substances below 1.400 in index, and only about five pages of these above 1.785. A set of liquids as described below, with index ranging from 1.411 to 1.785 in steps of 0.005, suffices therefore for all ordinary needs. For index ranges not covered in this paper the following will be helpful: West (12) has prepared liquids with index ranging from 1.78 to 2.06, using varying proportions of phosphorus, sulfur, and methylene iodide. Merwin (10) has prepared solid media with index ranging from 1.68 to about 2.10, containing three parts of antimony triiodide, one part of arsenic triiodide, and varying proportions of piperine. Barth (1) has described the preparation of mixed crystals of thallium iodide and thallium bromide with index ranging from 2.4 to 2.8. Harrington and Buerger (8) have used petroleum distillates for indices ranging from 1.35 to 1.46.

Previous Work

A great deal has been published concerning constituents of index liquids. For many years, many liquids, each with a different basic composition, were used to prepare a set (6). This allowed no gradation in index between two end members, and the index increment between adjacent members was irregular. Additional disadvantages of this type of set are (1) irregular changes in dispersion (2), so that the evidence of color fringes in oblique illumination tests is confused; (2)

different rates of evaporation of adjacent members of the set, so that the index of an intermediate liquid changes rapidly during the measurement. These factors are not so significant in double variation work where the properties of each liquid are accurately known, but they produce serious errors in oil immersion work with white light. Thus the tendency has been to use fewer liquids (4), carefully chosen to approximate an ideal series, from which intermediate members may be prepared by mixing the end members. In this way it is possible to prepare liquids whose components have about the same rate of evaporation, and whose indices remain constant during use.

TABLE I. DATA FOR PREPARING INTERMEDIATE INDICES

n_D at 22.0°	<i>n</i> -Decane Cc.	Government Oil Cc.	n_D at 22.0°	<i>n</i> -Decane Cc.	Government Oil Cc.
1.411	10.00	0.00	1.445	3.80	6.20
1.415	9.26	0.74	1.450	2.90	7.10
1.420	8.36	1.64	1.455	2.00	8.00
1.425	7.44	2.56	1.460	1.09	8.91
1.430	6.53	3.47	1.465	0.19	9.81
1.435	5.62	4.38	1.466	0.00	10.00
1.440	4.71	5.29			

End Members

The writers use the following end members to prepare liquids of intermediate index:

n-DECANE, $CH_3(CH_2)_8CH_3$, is very stable (?), colorless, $n_D = 1.411$ at 22.0° C., small dispersion, $d = 0.730$, b. p. = 174°, low volatility. (It is supplied by the Eastman Kodak Co., Chemical Sales Division, Rochester, N. Y. *n*-Decane, catalog No. 2405, 100 grams \$15.00. α -Chloronaphthalene, catalog No. 72, 1 kg. \$5.00. Methylene iodide, catalog No. 167, 100 grams \$4.50.)

MEDIUM GOVERNMENT OIL is very stable, colorless, $n_D = 1.466$ at 22.0°, $-dn/dt = 0.00035$, slight dispersion (?). (Leeds & Northrup Co., 4901 Stenton Ave., Philadelphia, Penna. Cost is about \$5.00 per gallon.)

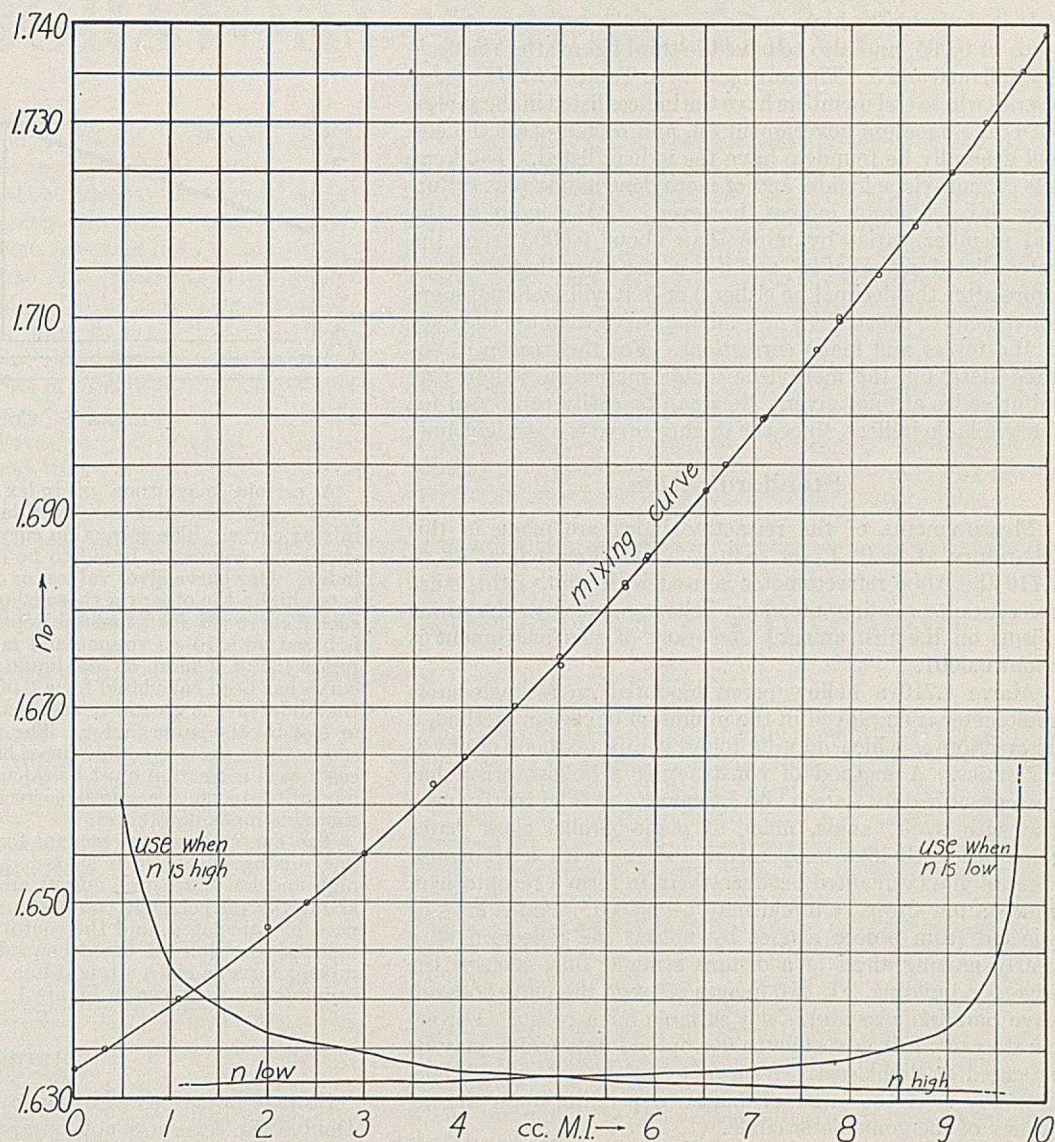
α -CHLORONAPHTHALENE, $C_{10}H_7Cl$, is stable, colorless, $n_D = 1.633$ at 22.0°, $-dn/dt = 0.0004$, moderate dispersion (9), $d = 1.191$, b. p. = 140–143°/20 mm.

METHYLENE IODIDE, CH_2I_2 , has a light brown color, $n_D = 1.739$ at 22.0°, high dispersion, $d = 3.325$, decomposes at 180°, $-dn/dt = 0.00070$. A few small pieces of c. p. copper (which is better for this purpose than tin) should be placed in all bottles containing this liquid to prevent decomposition and discoloration; the liquid should also be shielded from light as much as possible.

TABLE II. EXPERIMENTAL DATA

n_D at 22.0°	Government Oil Cc.	α -Chloro- naphtha- lene Cc.	n_D at 22.0°	Government Oil Cc.	α -Chloro- naphtha- lene Cc.
1.466	10.00	0.00	1.555	4.67	5.33
1.470	9.75	0.25	1.560	4.37	5.63
1.475	9.46	0.54	1.565	4.07	5.93
1.480	9.16	0.84	1.570	3.77	6.23
1.485	8.86	1.14	1.575	3.48	6.52
1.490	8.56	1.44	1.580	3.17	6.83
1.495	8.26	1.74	1.585	2.88	7.12
1.500	7.96	2.04	1.590	2.58	7.42
1.505	7.66	2.34	1.595	2.28	7.72
1.510	7.36	2.64	1.600	1.98	8.02
1.515	7.06	2.94	1.605	1.68	8.32
1.520	6.76	3.24	1.610	1.38	8.62
1.525	6.46	3.54	1.615	1.08	8.92
1.530	6.16	3.84	1.620	0.78	9.22
1.535	5.86	4.14	1.625	0.48	9.52
1.540	5.56	4.44	1.630	0.18	9.82
1.545	5.27	4.73	1.633	0.00	10.00
1.550	4.97	5.03			

FIGURE 1.
MIXING CURVES



METHYLENE IODIDE SATURATED WITH POWDERED SULFUR. The sulfur should be dissolved at room temperature and the excess sulfur filtered off, preferably with a suction device. This liquid has a light honey-yellow color whose index varies according to the degree of saturation with sulfur, and also according to the initial index of the methylene iodide. The index of solutions prepared by the writers has varied from 1.7848 to 1.7899. After the liquid is prepared a few grains of c. p. copper shot should be immediately added; it will soon turn black but will not affect the index of the liquid which remains perfectly clear for years.

Preparation

To determine the proportions of the desired intermediate members, one can construct a graph on a suitable scale, plotting index of refraction as a function of composition by volume. The proportions necessary to make any intermediate index can be read directly from this graph. Only in the case of an ideal solution will the mixing curve be a straight line (2). When making up whole sets it is convenient to have the composition scale read in absolute volume, instead of in per cent.

Two burets, each filled with one of the end members, may be used to mix the liquids. The correct volume of each liquid can then be put directly into storage bottles to await checking. When high accuracy in the third decimal place is not required, if large volumes are prepared (about 35 cc. to each bottle) and the volumes are measured very carefully, one can be reasonably sure that the indices are correct as read from the graph, without checking them.

The end members are *n*-decane and medium government oil for the range $n_D = 1.415$ to 1.465 . The mixing curve is a straight line; data (computed for a 10-cc. total volume per bottle) for preparing intermediate indices are listed in Table I.

The end members are medium government oil and α -chloronaphthalene for the range $n_D = 1.470$ to 1.630 . The mixing curve is a straight line; data are listed in Table II. Butler (4) gives excellent data for a similar series using kerosene fractions instead of government oil.

TABLE III. EXPERIMENTAL DATA

n_D at 22.0°	α -Chloronaphthalene Cc.	Methylene Iodide Cc.	n_D at 22.0°	α -Chloronaphthalene Cc.	Methylene Iodide Cc.
1.633	10.00	0.00	1.690	3.71	6.29
1.635	9.65	0.35	1.695	3.29	6.71
1.640	8.90	1.10	1.700	2.87	7.13
1.645	8.22	1.78	1.705	2.48	7.52
1.650	7.59	2.41	1.710	2.09	7.91
1.655	7.00	3.00	1.715	1.71	8.29
1.660	6.47	3.53	1.720	1.33	8.67
1.665	5.95	4.05	1.725	0.95	9.05
1.670	5.46	4.54	1.730	0.59	9.41
1.675	4.99	5.01	1.735	0.25	9.75
1.680	4.54	5.46	1.739	0.00	10.00
1.685	4.13	5.87			

α -Chloronaphthalene and methylene iodide are the end members for the range $n_D = 1.635$ to 1.735 . Since the mixing curve is not a straight line (Figure 1) these liquids are not "ideal solutions" but the authors' experience has shown that they are remarkably stable and form an excellent series (3). Experimental data for this series are given in Table III.

Methylene iodide and methylene iodide plus dissolved sulfur are the end members for the range $n_D = 1.740$ to 1.785 . The mixing curve is a straight line.

It is desirable to have the indices of the set graduated in steps of 0.005, and also to have the third figure after the decimal fall on 0 and 5. The mixing data in Tables I to III are for a series whose end members have the indices listed in the tables. *n*-Decane, medium government oil, and α -chloronaphthalene will generally be found to have the indices listed. Different lots of methylene iodide, and of methylene iodide plus sulfur, may have different indices, however. If the index of the end member varies by more than about 0.0005 from the values listed in the tables, and if it is necessary that the third figure after the decimal be either 0 or 5, it will be found more convenient to construct a new mixing curve than use the data in the tables and make corrections. For this reason, tabulated data for the methylene iodide-methylene iodide plus sulfur series are not given; they can be easily computed for the available indices, since the mixing curve is a straight line.

Standardization

Measurements of the refractive index are made in this laboratory at 22.0° C. in sodium light. For indices up to 1.710 the Abbé refractometer is used with white light, since correction to reading in sodium light is made with the Amici prisms on the instrument. Accuracy of this instrument is about 0.0001.

Above 1.710 a hollow prism mounted on a single-circle goniometer is employed in the minimum deviation method, a description of which may be found in any textbook of physical optics. A method of constructing a hollow prism has been described by Larsen and Berman (9). The writers used one with a 50° angle, made of plane-parallel glass plates cemented with high-temperature Piscein, with a triangular piece of glass cemented between them to form a receptacle in which a few drops of liquid may be placed. Plates may be selected from object glasses by noting the reflection at a nearly grazing angle of a distant straight line, such as the edge of a building. If two images are seen the plate does not have parallel faces and is not suitable for a prism. For accurate work it is advisable to use optical flats which may be procured at slight cost. With a 50° prism and optical flats, accuracy is of the order of 0.0001, depending upon the accuracy of the goniometer circle.

Temperature may be regulated by a water bath or by working in a constant-temperature room. It is especially necessary to have a temperature control when working with methylene iodide, since its dn/dt is high.

Corrections

The need for corrections may arise in mixing small quantities, or when old liquids are rechecked. For accurate corrections the amount to be added is calculated as follows:

We wish to prepare a liquid of index n_0 , whose proportions of end members *A* (low) and *B* (high) are x/y , and we have a liquid whose index n_h is too high, with proportions (which may be read from the mixing curve) of x_1/y_1 . An amount a of the low end member *A* must be added to this liquid to bring it to n_0 , so that $\frac{x_1 + a}{y_1} = \frac{x}{y}$ and $a = \frac{xy_1 - x_1y}{y}$. For example, if x/y , the desired proportion, is 4.5/5.5 (all proportions are on a 10-cc. basis), and x_1/y_1 , the actual proportion of the incorrect liquid, is 3.5/6.5, then $a = \frac{6.5 \times 4.5}{5.5} - 3.5 = 1.8$ cc. Thus 1.8 cc. of *A* (the low end member) are to be added to 10 cc. of the liquid of index n_h to change it to n_0 (see Figure 2).

Similarly, if we have a liquid whose index n_l is too low, with proportions x_2/y_2 , the high member *B* must be added in the amount $b = \frac{x_2y - y_2x}{x}$.

If considerable correction work is to be done, a correction curve may be drawn on the mixing curve graph.

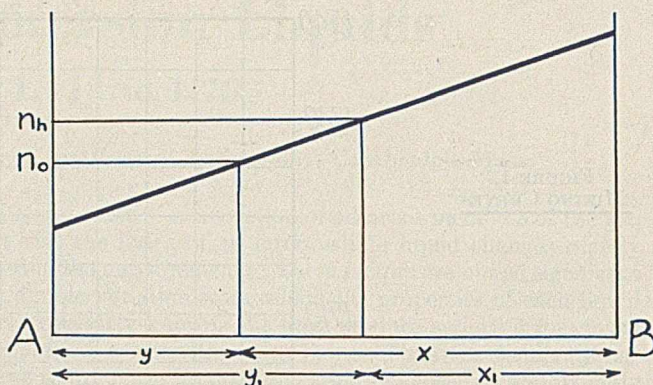


FIGURE 2. CORRECTIONS

A certain magnitude of index difference is selected—say, 0.001—and corrections are calculated for various parts of the mixing curve. The correction curve is plotted (Figure 1) and it shows the volume of liquid to be added to prepare any desired index. One curve gives values for correcting liquids whose index is too high; the other for those whose index is too low. In using such a curve two facts must be kept in mind: (1) The calculation is based on a 10-cc. volume and must be changed by an appropriate factor if more or less liquid is to be corrected. (2) The curve has been calculated for a 0.001 index difference; if the actual difference is greater or less than 0.001, a or b will be greater or less by the same factor. The multiplying factor is not the same where the correction curve has a steep slope and in these cases each correction must be calculated. Where the curve is of moderate steepness accurate correction may be obtained for considerable index differences.

The curves have the general form $xy = k$. For a straight-line mixing curve, such as *n*-decane and government oil, the high and low correction curves are similar and are symmetrical about the 50 per cent composition line. Hence only one side need be calculated, and the coordinates transferred to the symmetrical position on the other side of the graph. Where the mixing curve is not a straight line, the two correction curves are asymmetric, as shown in Figure 1.

Storage

Storage bottles should have as little air space as possible. Double air seals are not necessary if caps are kept tight. (Small square bottles of 15-cc. capacity, with glass applicators, and designed for this purpose may be obtained from the Central Scientific Co., Cambridge A, Mass. Catalog No. 66012; \$16.12 for two gross.) Each bottle should be shaken before using. The caps should never be interchanged.

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LABORATORY EQUIPMENT

WHATEVER part one plays in the conduct of research or the application of its results to any other form of activity, there must be a realization that without adequate laboratory equipment and supplies no scientific progress would be made. Indeed, there are several instances where advances have been arrested, pending the perfection of some new laboratory device. Too often those using such equipment fail to pause long enough to remember that research along other lines was required before suitable tools became available. Here and there, however, we find one who is really interested in instruments and apparatus of all sorts. Some research men have collaborated closely with instrument makers in the design and construction of instruments used by themselves and later made available to other workers.

It is with the thought that analysts and others would like to know something of the relationships between the makers of scientific apparatus and their own work, and particularly of the lines along which research proceeds in the effort to improve instrumentation, that the following articles have been assembled for this issue. The maker of scientific equipment is more than a merchant. Many manufacturers constantly seek the advice and assistance of the users of their wares and not a few support fundamental research either in their own establishments or under their auspices in scientific institutions. In this group of papers we make but a beginning in what will become an annual feature, providing it appears to fill a need. We want to do our part in increasing the appreciation which the users of apparatus should have for the efforts of those who undertake to supply them.

Monochromators and Auxiliary Apparatus

JOHN STRONG, California Institute of Technology, Pasadena, Calif.

BASICALLY new instruments depend on the development of new materials by chemists as well as the discovery of new properties, effects, and techniques by physicists. Tables I and II list some materials and physical effects which have been important in instrumental development in the past or from which we may expect future developments.

TABLE I. MATERIALS

Material	Properties, Uses, and Remarks
Methyl methacrylate polymers ^a	Not attacked by mineral oils Machines easily, does not dull cutting tools Thermoplastic, optical surfaces formed by molding Thermal expansion four times that of plate glass Electrical insulator as good as amber Very transparent ^b Can be cemented to form strong joints
Vinyl resins	Not attacked by mineral oils More impervious to gases than rubber, useful for vacuum hose ^c Aging more slowly than rubber, more inert toward ozone and ultraviolet light
Low vapor pressure oils ^d	Not attacked by mercury Useful for enamel and insulation for electrical conductors
Kovar and Fernico alloys (3, 12, 22)	Substitutes for mercury in vacuum diffusion pumps; no traps needed (31) Low vapor pressure waxes useful to line steel high-vacuum vessels (27)
Alnico alloy	Same expansion characteristics as glass for making glass-metal seals
Quartz	Strong permanent magnets Quartz wool-yielding filter material which does not sinter during ignitions ^e Quartz fibers, for suspensions in instruments (32) Reconstructed glass, yields odd shapes having low thermal expansion ^f
Glass	Fritted-glass filters Glass fabrics for filters Sealing glasses for sealing tungsten into fused quartz
Flexible Bentonite films	Inorganic parchment Mica substitute

^a Lucite, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del., and Plexiglas, Röhm & Haas, Philadelphia, Penna.

^b Spectrum range over which methyl methacrylate resins are transparent extends farther into ultraviolet than plate glass and, not so far into infrared. Transmission of a piece 0.5 cm. thick is 20% at 3000 Å. and again at 1.6 μ (19).

^c Koroseal tubing, Goodrich Rubber Co., Akron, Ohio.

^d Apiezon oils, manufactured in England and available through James G. Biddle & Co., Philadelphia, Penna. Synthetic organic oils, notably Octoil S, manufactured by Distillation Products, Inc., Rochester, N. Y.

^e Obtainable from Owens-Illinois Glass Co., Toledo, Ohio.

^f Corning Glass Works, Corning, N. Y.

New instruments will undoubtedly come from the ingenious combination of effects and materials already well known. Examples of this type of development are listed in Table III.

Finally, we have those developments which may be aptly described by the phrase "good engineering". The present article is concerned chiefly with the fundamental optical developments in astronomical and meteorological instruments with which the author is most familiar. These should be of interest to the reader because parallel developments in some chemical instruments may be expected. These developments, together with "good engineering", will increase the usefulness to chemists of all optical instruments now limited by their inadequate efficiency and sensitivity. Examples are monochromators (or spectrometers) used with either a photocell or thermopile in the visible, ultraviolet, or infrared part of the spectrum.

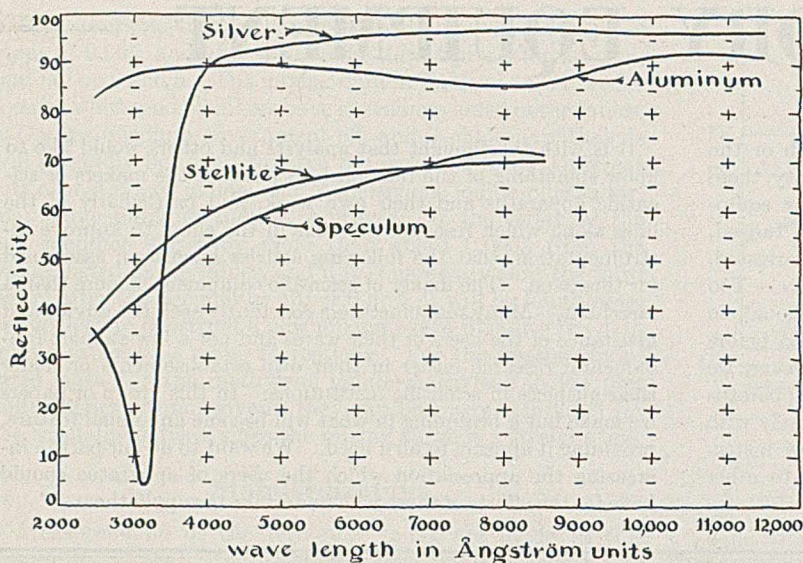
TABLE II. PHYSICAL EFFECTS AND TECHNIQUES

Technique	Applications
Electron optics	Improvements in electron diffraction apparatus Electron microscope Improved mass spectrograph (15, 16)
Isotopes	Enriched isotopes (example, H ²) Radioactive isotopes useful as indicators to follow course of chemical reactions
Spectroscopy (1, 24)	Infrared spectroscopy in chemistry for identification and analysis Colorimetry (18)
Vacuum technique	Low-pressure distillations for separation of hormones, vitamins, isotopes Delicate heat of reaction problems where use of high-vacuum, aluminized surfaces on containers and sensitive thermopiles will increase attainable precision

TABLE III. INSTRUMENTS

Name	Remarks
Electrometer tubes	Electronics
pH meter	Use of electronics techniques with glass electrode
Vacuum balance	Use of Alnico magnets
Packless and greaseless valves	Use of ductile brass and glass bellows ^a
Polaroid	Substitute for nicols (see Figure 5)
Colorimeters	Use of photovoltaic photocells
Fractionating columns	Employment of principles of Dewar flask
Super-high-pressure mercury lamp	Quartz-tungsten seals
Electric titrations	Use of electric eye

^a Metallic bellows obtainable from Fulton Syphon Co., Knoxville, Tenn., and Clifford Mfg. Co., Boston, Mass. For description of glass bellows see (2).



Credit, Prentice-Hall, Inc.

FIGURE 1. REFLECTIVITIES OF METALS USED FOR MIRRORS IN INSTRUMENTS

Surface Films by Evaporation

Glass is used for astronomical mirrors and lenses. The reflection coefficient of a glass surface is not ideally suited for either of these applications, being too low for the former and too high for the latter. But the reflection coefficient of the surfaces of mirrors and lenses is altered by thin surface films deposited by evaporation. This increases the efficiency in both cases; a thin film of aluminum (Figure 1) gives a glass mirror a reflectivity of approximately 90 per cent throughout the entire useful spectrum (27); a thin film of calcium fluoride on the surfaces of a lens eliminates 90 per cent of the transmission losses arising at the surfaces owing to reflection (29).

These surface films have been applied to increase the efficiency of monochromators. The use of evaporated aluminum films has eliminated the expensive roof prisms used in the early models of one double-quartz monochromator (see Figure 2). The use of evaporated fluorite films on this same double monochromator will more than double its transmission. The high gain resulting from the application of fluorite films is achieved because the light beam in this monochromator penetrates twenty quartz-air surfaces.

Double-Quartz Monochromator for Ozone and Water Determinations

The double-quartz monochromator, shown in Figure 2, is in use with the sun as a light source, to make routine analyses of the entire atmosphere for ozone and water vapor content.

For the ozone determination, which takes only 2 minutes, a sodium photocell in an evacuated quartz-glass envelope is used as a receiver. The photocell current is amplified with an electrometer tube (34). The intensity of the sunlight at 3050 Å. and 3110 Å. is measured. For the water vapor determination, which takes 5 minutes, the intensity of the ϕ -band at 1.15 μ is measured with a thermopile (10). It is noteworthy that the slits of the double monochromator remain the same for both determinations (0.075 mm., 0.003 inch).

Determinations have been made of the ozone in an absorption cell using the relative absorption at 3050 Å. to 3110 Å. Here a tungsten-filament lamp in a quartz-glass envelope serves

as the source of continuous radiation. (Tungsten lamps in quartz envelopes are supplied by the Phillips Laboratory, Eindhoven, Holland.) This source is useful from 2800 Å. to 3 μ . The voltage across the filament is regulated by a Raytheon regulator, the performance of which is expressed as follows (data taken with the output power adjusted to full load):

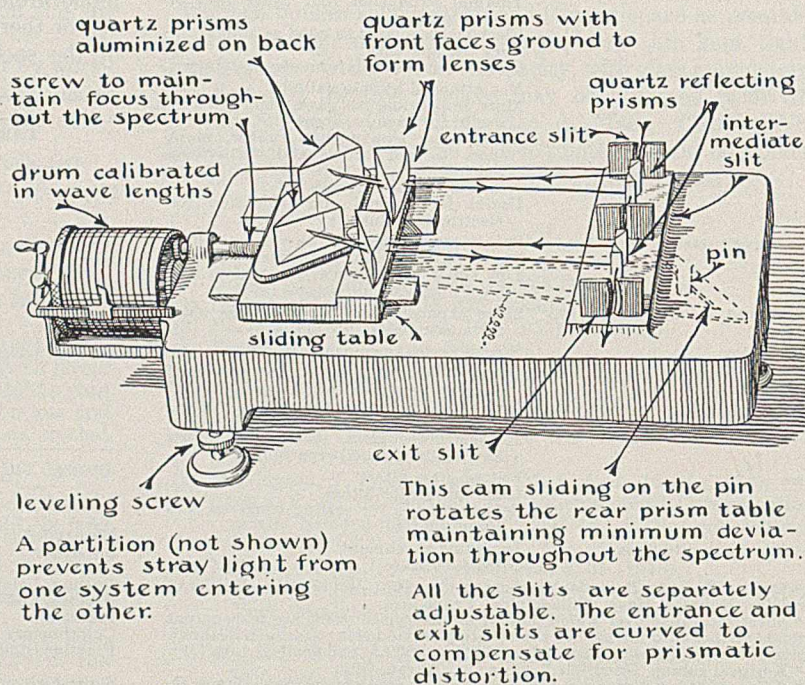
Applied voltage	70	80	90	100
Output voltage	114	116	116.0	116.0
Applied voltage	110	120	130	150
Output voltage	116.0	116.1	116.1	116.1

With the slits 5 Å. wide the response of the galvanometer at 3050 Å. is about 100 divisions. The ratio of the emission at 3050 Å. to that at 3110 Å., for the empty cell, is reproducible to one part in 1500. With this inherent reproducibility one can get an accurate determination of the ozone in the absorption cell.

The monochromator shown in Figure 2 and its accessories have been used for measuring the reflection of mirrors, the transmission of optical glass, the absorption of filters, and the spectral response of photoelectric cells. The use of a double monochromator is required for highly selective effects like the photoelectric effect, and its use in colorimetric work is recommended when highly selective receivers are employed.

TABLE IV. INDEX OF REFRACTION OF SYNTHETIC MATERIALS

Material	C	D	e	F	g
	6563	5893	5461	4861	4358
Fused quartz	1.4567	1.4587	1.4604	1.4634	1.4669
CaF ₂	1.4325	1.4338	1.4349	1.4369	1.4395
LiF	1.3906	1.3922	1.3930	1.3943	1.397
KCl	1.4870	1.4901	1.4929	1.4981	1.5043
KBr	1.5544	1.5590	1.5631	1.5709	1.5806
KI	1.6569	1.6655	1.6721	1.6853	1.7025
MgO	1.7337	1.7378	1.7412	1.7475	1.7550
Plexiglas	1.4856	1.4881	1.4902	1.4938	1.4992
Lucite	1.4916	1.4945	1.4967	1.5008	1.5064



Credit, Prentice-Hall, Inc.

FIGURE 2. HILGER-MÜLLER DOUBLE MONOCHROMATOR WITH QUARTZ OPTICS

Two prisms at left, shown here backed with reflecting coats of aluminum, were formerly backed with quartz roof prisms

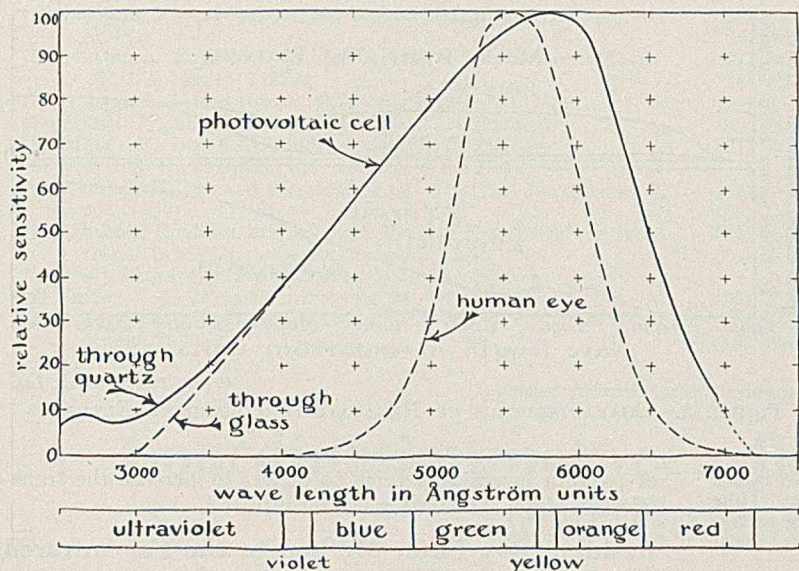


FIGURE 3. SPECTRAL SENSITIVITY OF PHOTOVOLTAIC CELL

Note that sensitivity goes to zero on long wave length side at approximately 7200 Å.

New Optical Materials

The infrared spectrum is being used now by chemists to identify compounds and to estimate the proportions in which they are present (1).

The growing of large synthetic crystals of the alkali halides was undertaken in order to supply prisms of new optical materials for infrared spectroscopy. Synthetic crystals of potassium chloride, potassium bromide, and potassium iodide were grown at the University of Michigan in cylinders 12.5 cm. (5 inches) in diameter by 12.5 cm. (5 inches) long (30).

To this list have been added two synthetic crystals, sodium fluoride and lithium fluoride, which are of importance in the ultraviolet and visible part of the spectrum (25). These two materials are important (4) because they simulate the properties of fluorite (see Table IV). The occurrence of fluorite in large size and quality suitable for optical usage is now very rare.

Another synthetic optical material now available (from the Norton Company, Chippawa, Canada) is magnesium oxide, which is useful in infrared spectroscopy for making shutters. Its transmission limit in the infrared is intermediate between that of quartz and fluorite (about 7.5 μ) and in the ultraviolet is 2300 Å. Magnesium oxide has a high index of refraction and a low dispersion (see Table IV). Its inertness toward the alkali metal vapors suits it for use as a window material for absorption cells to contain these vapors (35).

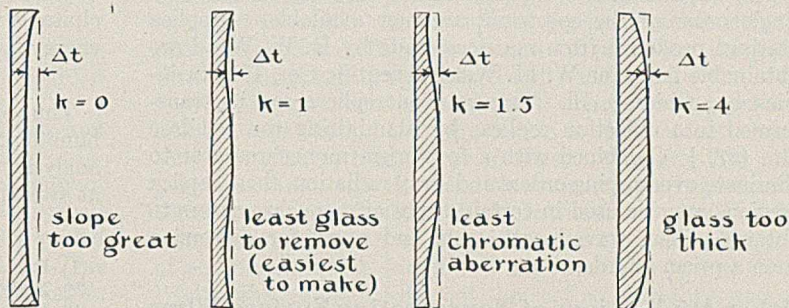
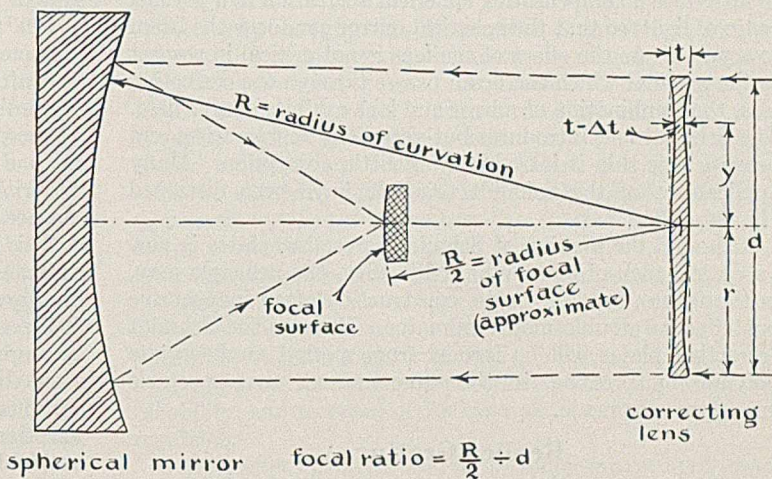
Synthetic thermoplastics (Lucite and Plexiglas) are now used for making unbreakable spectacle lenses and sun glasses and may eventually find extensive use in optical instruments (36). Their optical surfaces are formed by heat and pressure in polished molds without any of the customary optical working with abrasives and rouge.

Radiation Receiving Devices

Photocells are not treated here (they are adequately discussed elsewhere, 18) except to point out that the sensitivity of the photovoltaic type vanishes completely at about 7200 Å. (see Figure 3).

Improvement of the sensitivity of thermopiles and other radiometric devices has been the object of many investigations (33) and work is now being done to improve their performance. There seems at present no possibility of making "order of magnitude" improvements in the sensitivity of this type of instrument, which measures a flux of radiant energy, because the delicacy of the measurements is now limited by the effects of Brownian motion. Recently, however, a novel method has appeared for photographing in the infrared spectrum out to 9 μ (5-7, 17, 37). Because, in principle, this new method integrates the flux of energy over the time of exposure, it is not inherently limited by the effect of Brownian motion and we may reasonably expect that it or some modification of it may be developed which will yield an instrument of great sensitivity.

But without radical developments in radio-



These curves may be put on either or both sides of the lens provided the value of (t-Δt) is maintained.

$$\Delta t = \frac{y^4 - k r^2 y^2}{4(n-1)R^3}$$

k = a constant between 0 and 4
n = index of refraction of lens material

Credit, Prentice-Hall, Inc.

FIGURE 4. DIAGRAM OF SCHMIDT PRINCIPLE AS APPLIED TO AN ASTRONOMICAL TELESCOPE

Above. Parallel light from the right is modified by the correcting lens so that the spherical mirror at the left focuses it to a point. Below. Various shapes that may be given the correcting lens and formula describing their contours

metric instruments we can expect definite advances in the technique of working with the instruments now available.

For example, to measure with a thermopile most effectively the energy emergent from the slit of a monochromator (especially as applied to infrared spectroscopy) it is necessary to form a reduced image of the exit slit on the thermocouple and at the same time increase the solid angle within which the thermocouple is irradiated. At present an elliptical mirror is used, giving a fivefold reduction in the image size and an increase of angle of irradiation to a cone of 100° diameter (9, 20). The thermopile is at one focus of the elliptical mirror and the exit slit of the monochromator is at the other focus. The elliptical mirror is not completely suited for this application, for although the image is definite on the optical axis the imaged ends of the slit which lie off the optical axis exhibit coma (8). This coma necessitates the use of an oversized thermopile receiver. The technique may be improved by applying the Schmidt principle instead of an elliptical mirror. Thus, without any sacrifice in the solid angle of irradiation, the area of the thermopile receiver may be reduced three- or fourfold with an attendant doubling of the thermopile sensitivity.

The Schmidt Principle in Optical Design

The Schmidt principle in optical design is described in a recent issue of the *Scientific American* (11). The Schmidt principle involves the use of a lens positioned at the center of curvature of a spherical mirror (Figure 4). The lens is figured to introduce a compensating spherical aberration in a parallel beam of light, so that the spherical mirror can focus the beam to a point. As the effect of the lens is not critical in respect to the angle at which the beam passes through the correcting lens, the combination of mirror and lens exhibits a large field. The Schmidt lens introduces but very little achromatism and because it is thin it introduces but little absorption. Many applications of the Schmidt principle have been discussed (11, 23, 26).

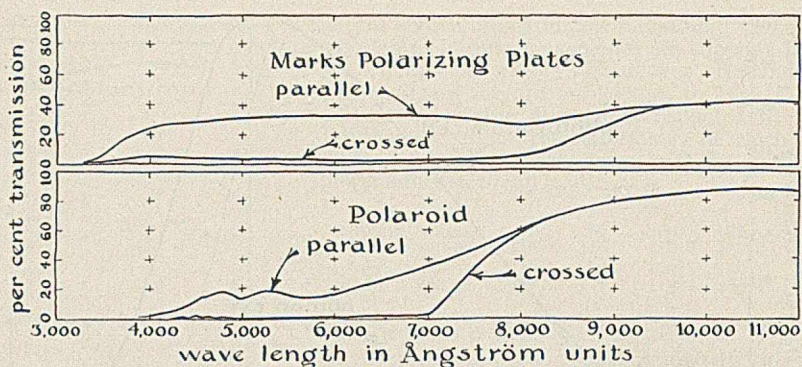
Although the making of Schmidt correcting plates is now an optical task of considerable difficulty, the principle may, in the future, be used in the construction of an inexpensive but effective double monochromator. Perhaps the Schmidt correcting plates will be formed from methyl methacrylate polymer by pressing, in the same manner that spectacle lenses are now made.

Replica Gratings

Excellent replica gratings (both reflection and transmission types) which throw a large fraction of the incident light in a single order of the spectrum, are now available. [Replica gratings produced from matrices made by R. W. Wood are obtainable from the W. M. Welch Scientific Co., 1518 Sedgwick St., Chicago, Ill. Transmission replicas can be transformed into reflection replicas by aluminizing the cellulose film (27).] Combined with a fore-prism monochromator to eliminate overlapping orders and stray radiation, these replica gratings may be used in certain types of monochromators to obtain a linear wave-length scale and a greater dispersion than a prism affords.

Other Applications of Nonreflecting Surface Films

Nonreflecting surface films of fluorite and other substances are deposited by evaporation on optical surfaces primarily to make optical instruments more efficient. In addition, these nonreflecting films eliminate halations and the thin veil of background light produced in a lens by internal reflections. Many applications of these films are possible, wherever surface reflections from a transparent optical body are to be eliminated. Promising among them are the application of non-reflecting surfaces of fluorite to decrease the reflectivity of residual-ray crystals in the near infrared and the application



Credit, Prentice-Hall, Inc.

FIGURE 5. CHARACTERISTICS OF HERAPATHITE POLARIZING ELEMENTS

of paraffin layers 20μ thick to quartz to increase the transmission of quartz lenses in the far infrared.

Residual-Ray Filter Method in the Far Infrared

The residual-ray filter method uses successive reflections from crystals to obtain monochromatic bands of infrared radiation. The method affords, in effect, a monochromator. Wave-length bands from 6.7μ to 150μ are thus obtained (8, 14). A new arrangement of the residual-ray apparatus using a band of radiation at 8.8μ affords a pyrometer particularly suited for determining surface temperatures in the range -100° to $+100^\circ$ C. (28). The band of radiation used in this pyrometer as the thermometric property lies at a position in the infrared spectrum where water vapor is very transparent. Accordingly, no corrections for absorption in the optical path of the pyrometer are ordinarily required. With the pyrometer one can measure the temperature of surfaces without interfering with heat loss by radiation and convection or with surface heating.

This instrument was intended for astronomical (sun, moon, star, and planet temperatures) and meteorological (ozone, air, tree, grass, and ground surface temperatures) applications but it is possible that it will find other applications. Fitted with appropriate crystals, the residual radiation falls exactly on the ozone band lying in the infrared spectrum at 9.6μ and the instrument so arranged is being used daily to measure the absorption of sunlight by the ozone of the atmosphere. Using a residual-ray band at 6.7μ obtained with calcite crystals, there is a linear relationship between the square root of the water vapor in the optical path, τ , expressed as centimeters of precipitable water, and the produced fractional absorption of the energy in the residual-ray band, A :

$$A = 4.4 \sqrt{\tau}$$

Thus one may determine, with the apparatus, the absolute humidity. The above relation on which this "chemical analysis" is based is valid only at ordinary temperatures and at atmospheric pressure (13, 21, 38).

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Coordination between Instrument Maker and Research

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THE close integration that may exist between instrument maker and research laboratories is well illustrated by the experience of this company. The men who founded the organization 25 years ago were skilled technicians who had been associated exclusively with experimental groups. It was natural, therefore, that their independent activities should from the start have consisted in the construction, according to sketch or published paper, of fine instruments and equipment for governmental, educational, and industrial laboratories. Many published contributions have rested strongly on a specialized apparatus carefully constructed by this establishment.

This policy of close contact and collaboration with organized research groups, although greatly extended and varied, has remained essentially the same through the years. In a large new plant, generously equipped with standard and highly specialized tools, construction proceeds at any one time on a number of different instruments designed to fill special research needs. Scientists from governmental laboratories and from schools in the vicinity of Washington, D. C., frequently avail themselves of facilities not to be found elsewhere. The present new plant has 20,000 square feet of floor space. The organization is divided into several closely knit sections, the principal ones being the technical and development section and the fine tools division.

The rapid tempo of scientific development requires incessant scrutiny and study of the literature and contact with leading laboratories.

New instruments and new designs of old instruments find their fitting counterpart in new materials of construction, machines, and methods. Skill, resourcefulness, and economy of manufacture alone no longer suffice to meet the demands of an exacting scientific world, but the fabrication must be guided by and conform to inexorable theoretical requirements. This aspect is taken care of by the highly trained technical section, which is closely integrated with the production department. It is not unusual for the construction of an instrument and its proper employment to be conditioned by exacting mathematical relationships.

Coordination with and service to chemical and physical research laboratories are, however, hardly complete if they stop at the point of making specialized instruments. The plant and skills of the organization are therefore directed toward the manufacture of standard instruments and routine laboratory equipment. In all these operations the same type of skilled mechanic is employed as in the special instrument con-

struction. The standard apparatus is mainly for testing, for control, and for analysis, and fabrication is of metal, glass, or other material.

Services

It may be thought that the product of an instrument company is nothing more than a home-made gadget in fancy dress. This is far from being true. Into each instrument enter desirable and indispensable values that result from much thought, planning, combined skills, and the application of special tools and methods. The product of a reputable instrument company will give more accurate, rapid, and certain measurements and results than the comparative makeshift of the hurried and unspecialized laboratory worker. A case recently came to notice of the loss of time and ruination of experimental work caused by a home-made thermoregulator. The construction seemed relatively easy, but unexpected difficulties of fouling of the mercury, breaking of the thread, etc., arose which are to be contrasted with the simplicity, reliability, and accuracy of, for example, a metastatic thermoregulator.

Besides offering substantial improvements over home-made equipment, a further type of service consists in the production of instruments and apparatus that the average shop is scarcely equipped to make. An example is the preparation of fused glass absorption cells with guaranteed plane parallel ends, which requires highly specialized skill and tools.

A third type of service consists in a more economical and efficient construction than is possible in the average laboratory, yet with at least equal accuracy. Recently the company was faced with the task of constructing a respirometer according to a published design. It was found possible after suitable consideration to prepare a very much simpler piece of equipment which functioned the same and just as effectively; the simplifications cut the cost considerably, and resulted, in fact, in putting the instrument on the general market.

A further contribution of value consists in making generally available, very shortly after publication or announcement, instruments and equipment that have obvious merit. There have been several instances in which a demand for an instrument, promptly proclaimed after publication, was just as promptly met. In this way the findings and accomplishments of research laboratories come into general use a long time, years perhaps, before they would without the service rendered by the modern instrument company.

The company has found it desirable to sustain cooperative fellowships and research undertakings in connection with instrumentation. Two such are in operation at present. The main objective of one of these has been to develop the most scientific method of use and most efficient design of an important instrument for fine size analysis, which is manufactured by the company. This fellowship has been unusually fruitful and has resulted in fundamental findings. The second research is in the automotive industry and has to do with the study of the various conditions of use of engine indicators so as to develop their widest utility.

Costs

The undeserved penalty of good appearance is sometimes the impression that the cost has been polished up, so to speak. As far as established instrument making is concerned, this is not true, nor can it be under competitive conditions. The notion may be based on a hurried and fallacious method of accounting. One cannot estimate the cost of an instrument, any more than anything else of use value, on the basis of the value of the component materials. There are items to be

considered of initial technical investigation, experienced engineering design, and skilled machine construction, all of which lead to a more efficient, more durable, and more presentable article than in the absence of these services. One need hardly mention also the usual economic items of capital investment, obsolescence, overhead, salary, etc., which should be taken into account when an investigator attempts his own construction. Finally, one may note the saving of valuable time and effort, as far as the real objective of the investigator is concerned, when he has before him a highly satisfactory instrument, ready for use.

In conclusion, the instrument maker is interested in the problems of the investigator and research worker. In fact, he has to be, for it is only on the basis of the broadest knowledge of the requirements of the laboratory that he is able to render a fitting service. In this sense, he welcomes the inquiries and interest of the investigator, and he is prepared fully to cooperate with him.

The scientific research world has been implemented thereby in a manner that has created more accurate results and broadened research horizons beyond the fondest dreams considered possible only a few short years ago.

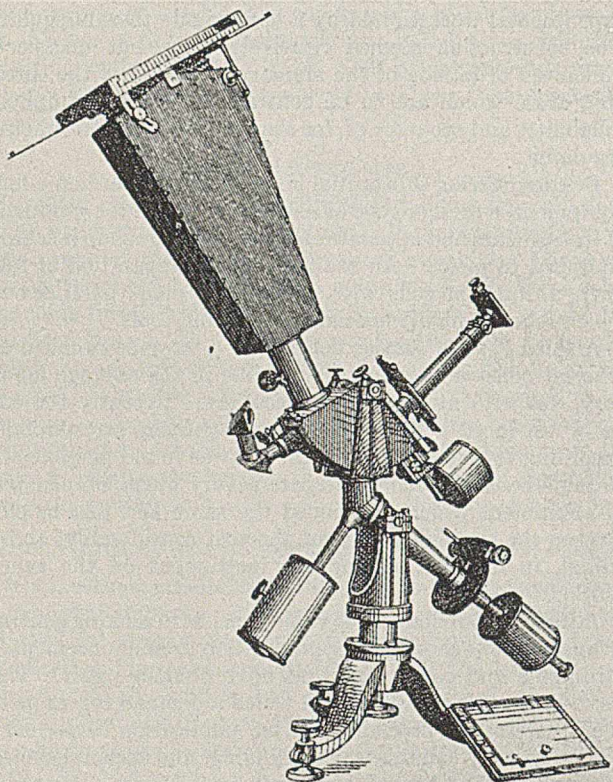
Spectrograph Design and Its Problems

J. W. FORREST, Bausch & Lomb Optical Co., Rochester, N. Y.

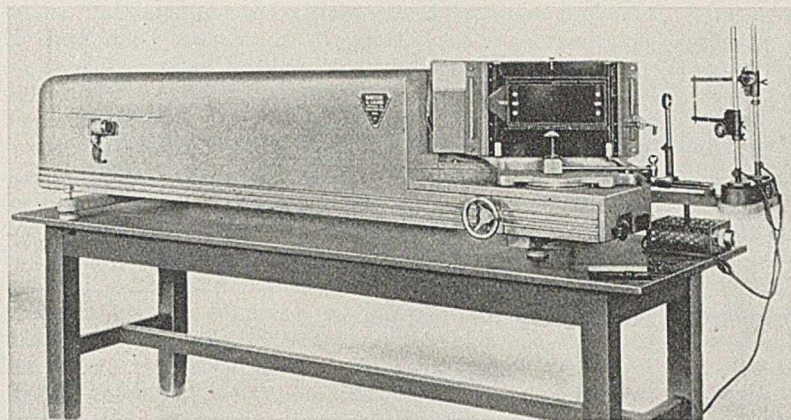
OPTICAL instruments of various kinds are extremely valuable in chemical analysis. Microscopes, colorimeters, polarimeters, saccharimeters, and refractometers are extraordinarily useful tools for the identification of unknown substances or for the determination of the degree of concen-

tration of known substances. They are valuable for the kind of information they may supply, for the speed with which required information may be obtained, and for the degree of accuracy obtainable in comparison with other methods. While comparisons are dangerous, one is tempted to say that the spectrograph is more useful to the chemist than all the above-mentioned instruments. It is deplorable that there is no word which includes both spectroscope and spectrograph. In spite of the fact that the spectrograph with its permanent photographic record of both visible and invisible portions of the spectrum is the most commonly used, the spectroscope is too useful to be ignored. The reader should interpret the word "spectrograph" in this article to include spectroscope and spectrometer unless it is obvious that only the photographic form is meant.

For years the spectrograph was the physicist's most powerful tool in his search to unravel the secrets of the construction of matter, and unaided it led him to the necessity for recognizing orderly arrangement in the complex structure of the atom. Lockyer in 1873 for the first time advanced the theory that changes in line spectra, due to rise in temperature of the source, could be explained by the breaking up of the atom just as the transition from band spectra to line spectra may be explained by the dissociation of the molecule. The chemist soon recognized its power to reveal the composition of unknown materials and the astrophysicist made it his basic instrument. It has revealed the fact that the universe is apparently made up of the same chemical elements that compose the earth. Without the spectrograph it is impossible to imagine how we could have acquired any information whatever about the composition of heavenly bodies except what might have been gleaned from the occasional meteors which reach the earth. Matter, emitting or absorbing radiation which the spectrograph can analyze into its component wave lengths, reveals not only its identity but much about the state in which it exists. Distance is immaterial, provided enough light reaches the observer to affect the eye or the



STEINHIL SPECTROGRAPH OF 1894



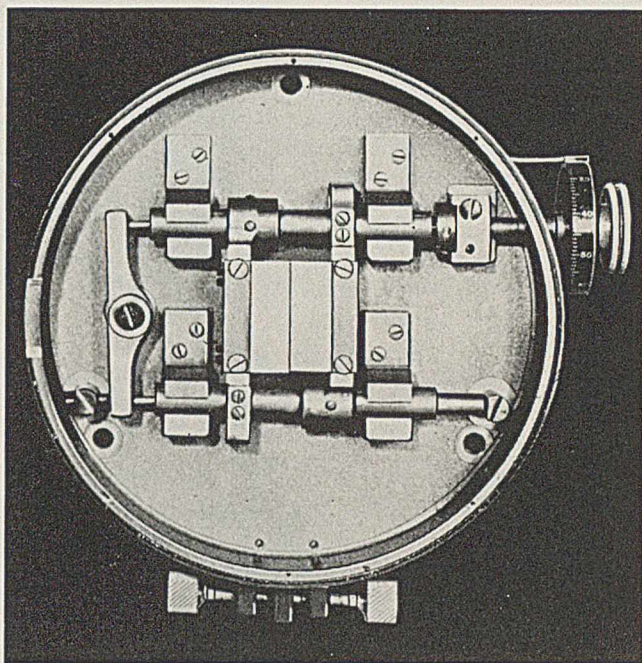
MODERN QUARTZ SPECTROGRAPH, LITROW TYPE

utmost efficiency in operation. They must be adjusted in the factory and must maintain that adjustment during transportation and over a long period of possibly daily use. The operator must learn the mechanical technique of operation through the reading of some rather simple directions for use. In his hands the instrument must be dependable in spite of a certain amount of abuse, the worst of which is probably experienced when it falls into the hands of one who cannot keep his fingers off the adjustments. Finally, it must be powerful enough for the work required without excess power which leads to excessive cost and difficulty of operation.

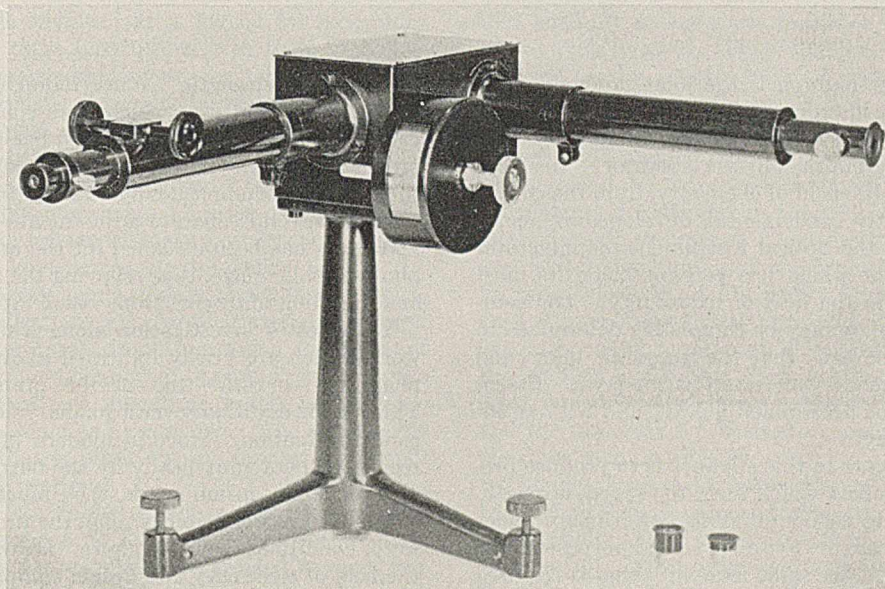
photographic plate. The arc on the laboratory table or the distant nebula tells the same story.

So long as the instrument was useful only for qualitative chemical analysis, it was not of great value in the laboratory. Its major function as a qualitative instrument was in the identification of trace elements, the concentration of which was so weak that they defied ordinary chemical analysis. This period of spectrographic science dates from the time of Bunsen and Kirchoff to about 1875 when Lockyer proposed its use for quantitative work. He was followed by Hartley in 1884 and Pollok, Leonard, and de Gramont about the turn of the century. These men laid the basis for quantitative analysis and since their time the science has been progressing so swiftly that one hesitates even to suggest what may represent the ultimate limits of its usefulness.

In the early days of spectroscopy, the investigator usually had to make his own instrument. In the graduate school of a university, where time is usually more abundant than money, this condition could be tolerated even though it yielded an instrument operable satisfactorily only by the one who designed and built it. For the modern industrial laboratory, where time is the most valuable element and the results of the new investigation or of the routine analysis are the all-important objectives, the design of instruments must offer the



SLIT MECHANISM FOR LARGE SPECTROGRAPH



WAVE-LENGTH SPECTROMETER FOR VISUAL REGION

ditions and determine the optimum for a particular problem. He must study the relations of inductance and capacity, and eventually he must be thoroughly acquainted with the influence of one element upon another when both are volatilizing at the same instant. Practically in the present status of spectrographic science this has become a limiting factor and is a problem, which, when satisfactorily solved, offers promise for greater accuracies than can now be attained. Physicists and chemists in various fields are seeking the answer and it is hoped that major advantages will come from these investigations. The Bausch & Lomb Optical Company is making the necessary plans to support a fellowship at the Massachusetts Institute of Technology, the sole purpose of which will be to investigate the basic phenomena occurring in sources of energy where metallic vapors exist. Such investigations as these provide the basis upon which the designer can rely to build into his mechanical, optical, and electrical structures new features which will increase the reproducibility and hence the reliability of the results. As is true with any growing science, many problems of fundamental nature are still unsolved and the solution of any one usually makes it necessary to redesign existing apparatus or to devise new apparatus to meet the need.

Another phase of design which is not often given enough consideration is that of the study of the materials in the optical structure. Today most of the work is done in the ultraviolet or in the infrared region where suitable materials are few and expensive. The designer must utilize these materials to the utmost and must be sure at all times that they

are neither inferior in quality nor poorly fabricated. This means that he must be, to a certain extent, a student of crystallography in order to design and test systems which will function to the greatest advantage and convenience of the user. Quartz, calcite, fluorite, and rock-salt all enter the optical structure in one place or another and each offers its own problem in the design of specific optical systems. Quartz and calcite are birefringent and the former is rotatory also. Each requires special forms of computation to produce the desired results. Rock-salt is hygroscopic and offers its own peculiar problems. Only careful selection of material and careful handling of the varied optical constants can achieve useful results.

The combined efforts of many individuals are usually necessary to bring about material advances in any scientific work. In this brief account of developments in a single group of instruments, we see the designing engineer drawing his data from industrial and educational laboratories, combining these with his own knowledge of materials and design, and turning back instruments which contribute to increasing accuracy and additional achievements in many fields. The chemist, the physicist, the astronomer, the biologist, the geologist, the criminologist, and even the dealer in scrap metal in one way or another derive profit from the joint efforts of the laboratories and the instrument designer. Whether it be the composition of the universe millions of light years away, or the composition of the discarded bit of alloy in the junk yard, the spectrograph is capable of yielding the answer quickly and in a manner that is not usually subject to dispute.

The Laboratory Supply House

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LABORATORY supply houses are not merely dispensers of merchandise. Very few people realize how far their service extends and how vital is their contribution to industry, science, and economic welfare. It is logical for laboratory workers to approach these institutions with their problems, for where else can such complete service be rendered? A staff consisting of chemists, physicists, bacteriologists, instrument makers, and glass blowers, with instrument shops, glass-blowing shops, literature references, files, and similar facilities, is maintained at great expense by these firms. The older laboratory supply houses are libraries of information, whose functions are to render willingly a practical service to those in the field who have perplexing problems.

We read of the progress made in developments of plastics, synthetic textiles, medications, etc., better materials produced at lower prices, better methods of preserving foods, purer foods, sanitary control, eradication of disease, increase in the span of life, creation of new industries, better homes, etc., all as a result of achievements by research. The significance of the contribution that is made by the laboratory supply firm is often overlooked entirely.

It is true that in the brain of the researcher ideas are created, but mental visions do not always become practical processes unless tools are developed or created to carry out the ideas. For instance, a scientist evolves a plan for manufacturing a new product. He finds during the development

stage in the laboratory that he needs a special piece of apparatus. Since he is no artisan and has no facilities for fabrication, he approaches a laboratory supply house, as a source that not only makes these available but is able to conceive what he has in mind. Thus, frequently the task becomes the problem of the artisan assigned to construct an apparatus that will finally carry the problem to success.

Some years ago, this company was approached by a scientist who had an idea that the product his firm was manufacturing could be improved in quality and produced more efficiently. He outlined his theory and explained the existing methods and processes, and in a short time a thorough knowledge and understanding of the problem were acquired by the artisans delegated to the task. It was necessary to devise an instrument for actual tests and control of the materials, and after many months of investigation and experimenting, the problem was solved most satisfactorily. The firm, thereafter, was able to produce a better and more uniform product, and the undertaking resulted in many economies and a tremendous increase in sales.

Though the role that was played by the laboratory firm has undoubtedly long been forgotten, it certainly contributed substantially and in many ways to the benefit of the industry. This is but one of numerous incidents where the research worker and industry have benefited by the scientist's cooperation.

Analytical and Microbalances

A. W. AINSWORTH

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VERY few users of analytical balances realize or appreciate what has been done in recent years to improve reliability and reproducibility particularly at the higher sensitivities, and it is the intention of this article to state some of the difficulties encountered in the efforts to give the chemist a more reliable piece of apparatus than has heretofore been obtainable.

The beam, being the most important part of a balance, has of necessity been given the greatest amount of attention, and during the investigation of the behavior of balance beams under varying conditions such as load, temperature, and humidity, some very interesting information has been obtained. The first important discovery was the great variation in the material (aluminum alloy) commonly used for balance beams, and after definitely determining these peculiarities, the finish was found to have a wide bearing on the

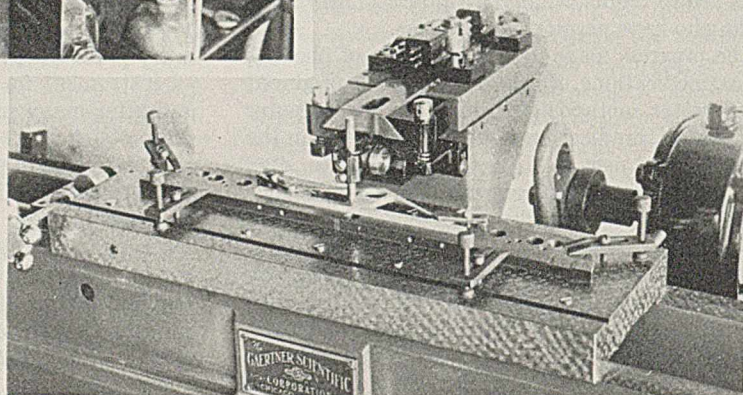
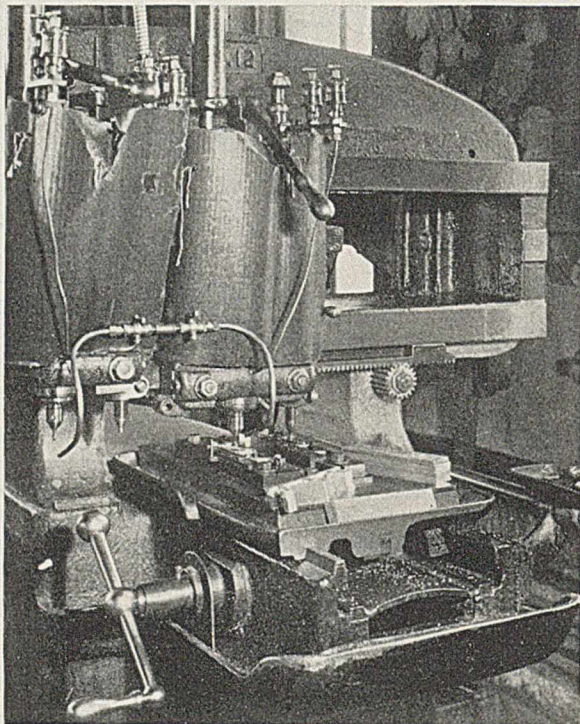
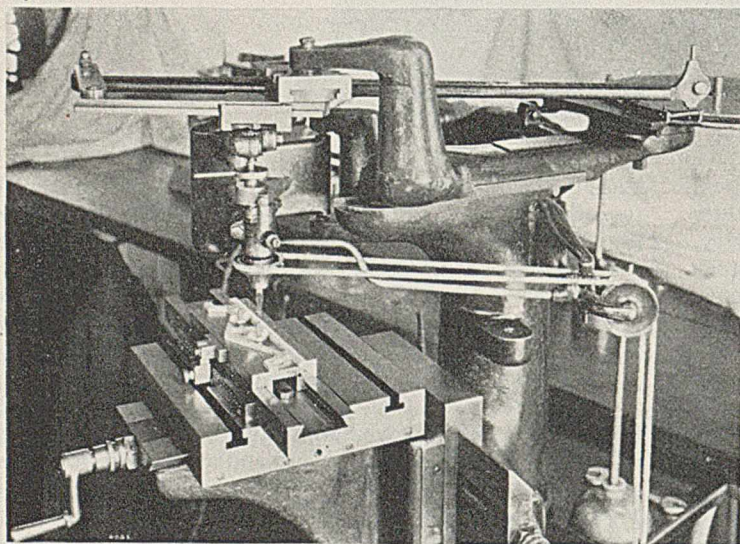
performance and stability of the beam. Various alloys, while varying from one another only in very small amounts, were discovered to behave in entirely different manners. Strains due to the mechanical working of the material itself were of major importance and it was during the above-mentioned investigations that a method of normalizing this material was discovered.

The actual production or machining of the beam from the blank material brought its attendant complications which were more easily surmounted but, nevertheless, contributed to some of the peculiar behavior of the beams. As these problems were gradually approached and carefully investigated, the question of the knife edges in relation to their attachment to the beam came to the fore, and it was during this particular investigation that a great deal was learned not only of the proper fitting of the edges to the beam but of

FIGURE 1. PRODUCTION OF BEAMS FROM BLANK MATERIAL
(upper right)

GRADUATING OF STANDARD ANALYTICAL BALANCE BEAM
(lower right)

ENGRAVING OF NUMBERS OF BALANCE BEAM (left)



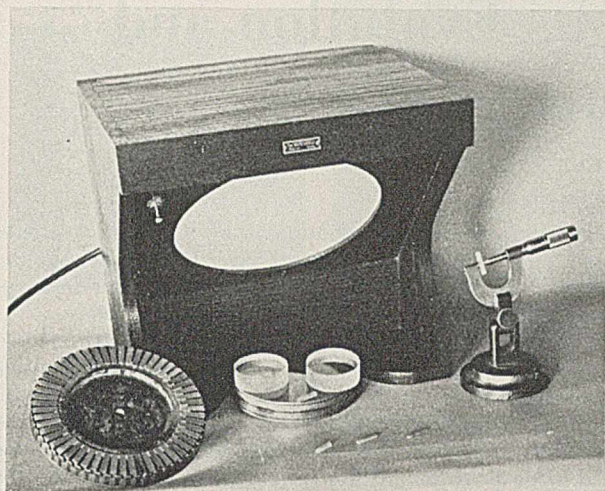
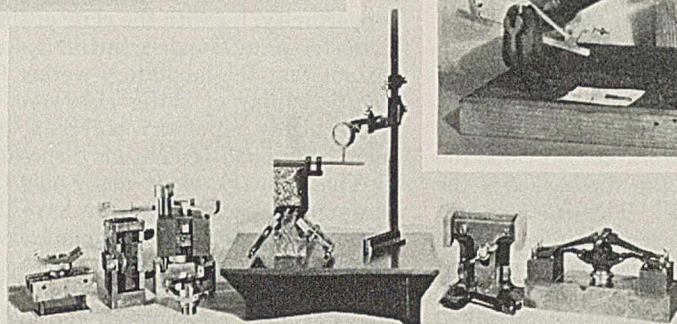
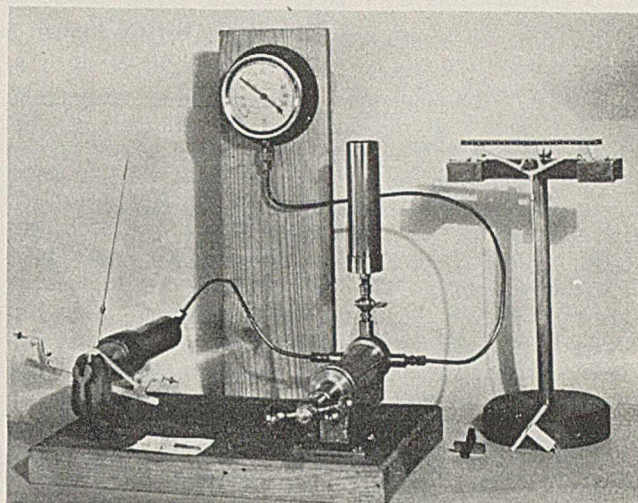


FIGURE 2. FIXTURES FOR GRINDING AND TESTING AGATE EDGES AND BEARINGS (left), SETUP FOR DETERMINING FIT BETWEEN AGATE EDGES AND BEAM (right), AND JIGS AND FIXTURES FOR CHECKING PARTS (bottom)



the proper production of these edges. The grinding of the infinite knife edge, which is the actual support medium of the load suspended by the beam, is important, as the behavior of the beam is due very largely to the accuracy of this edge. Various methods of grinding and polishing this contact edge have been tried, with the result that balances have been constructed of a higher sensitivity and capable of handling heavier loads than have previously been possible. The various devices used in determining the qualities and qualifications of the agate knife edges were rather ingenious and original. Other important problems that presented themselves were the accuracy of the graduations and the numbering of the beams. It was found that where the numbers were stamped, the time required to obtain a stable beam was many times longer than when the numbers were engraved by actually removing the material with a revolving cutter.

To take advantage of these new refinements in the production of balance beams, more care was necessary in the design, production, and assembly of the parts used in connection with the beam-releasing mechanism. Many conditions not conducive of smooth and accurate operation of this part of the balance detracted from the improvements obtained in the beam; therefore, manufacturing jigs and fixtures had to be made to much closer tolerances than had been previously used. Machining operations changed, and improved inspection methods such as had been used only by the manufacturers of high-grade tools were adopted. These improvements, together with the use of metal cases, have made it possible to maintain close adjustments and alignment of the various parts of the balance.

The photographs of Figure 1 show (upper right) the production of beams from the blank material. The method adopted assures that a minimum amount of machining strains will be in the beam; two separate cuts are made—a

rough cut and a finishing cut. The graduating of a standard analytical balance beam is shown at lower right, a highly accurate graduating machine being automatically compensated for temperature; and at left, the engraving of the numbers of the balance beam.

Figure 2 shows (upper left) some of the fixtures for grinding and testing the agate edges and bearings used on the balance beams; (right) setup used to determine the closeness of the fit between the agate knife edges and the beam; (lower) jigs and fixtures for checking the various parts of the releasing mechanism, drop levers, etc., as well as a setup for continually inspecting the accuracy of the work as it comes from these fixtures.

A great deal of work has been carried on in connection with the development and production of a successful microbalance and at this time a number of microbalances are in use whose performance is extremely gratifying. The information that is being accumulated in reference to their performance under varying degrees of temperature, humidity, and barometric pressure will result in making available to the chemist balances capable of carrying greater loads at higher sensitivity with greater availability and reproducibility than has heretofore been available.

In connection with the investigation of the performance of microbalances, a very interesting development has been carried on in an effort to read more accurately the deflection of a microbalance beam, an entirely new principle being used. While this has not proved entirely satisfactory as a reading device, it has, nevertheless, brought to light some very interesting facts, heretofore unknown, regarding the actions of a balance beam. It is believed that the knowledge that has just been gained will lead to a more thorough understanding regarding the sometimes erratic performance of microbalances and will have its effect on the future development of the standard analytical balance.

Laboratory Apparatus, Its Evolution and Development

WM. B. WARREN, Fisher Scientific Co., Pittsburgh, Penna.

LABORATORY appliances, starting with the early crude forms shown in the accompanying illustration, have been improved in keeping with the advance of the various sciences to which they are so necessary. In fact, advance has been mutual; new discoveries in the sciences have led to the development of new laboratory tools, and in many cases, the development of a new tool has paved the way for great scientific advances.

The early chemist needed to be a versatile soul, for he had not only to plan and carry out his laboratory work, but also to be his own instrument maker. Chemist, machinist, glass blower, jack-of-all-trades—who knows what might have been the result had such great minds as those of Bunsen, Liebig, Lavoisier, and Kekulé been free to concentrate on the significant matters which they were uniquely endowed to pursue?

With the growth of early scientific work abroad came the inevitable advent of specialization and the appearance of the early instrument maker and glass blower. These men worked closely with those in the centers of scientific investigation and often played important roles by developing new apparatus or improving the older forms.

Since so many of the original workers in this country had studied abroad under the older chemists, it was only natural, when they started to carry out their studies or to teach here, that they sought the equipment which they had used abroad. Laboratories were still rarities, and those who supplied their requirements were mostly importers.

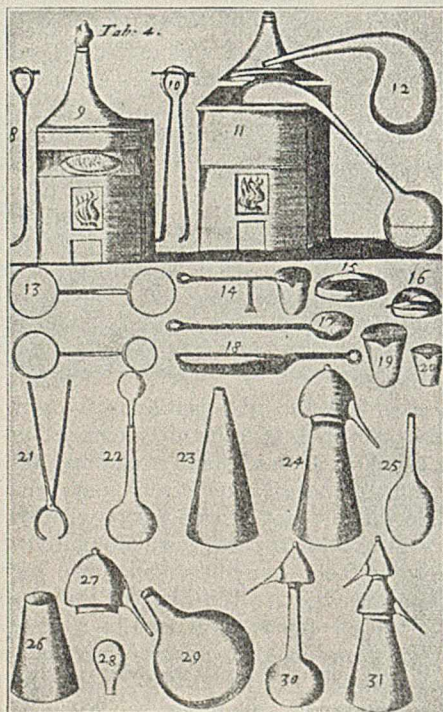
The advent of the Great War found us not only without a significant chemical industry, but also without an adequate

domestic source of laboratory apparatus with which to create such an industry. It was during the early war days that the American chemical industry, together with the American apparatus industry, began to develop to what it is today.

In the initial rush of war preparations, there was time to do nothing but manufacture prototypes of the foreign apparatus. No attempt could be made to introduce improvements; production was all-essential. Gradually production was stepped up until urgent needs were fulfilled and there was time available for development work. Good use was made of this opportunity, for with the close of the war not only had a new industry been founded, but it had rooted and grown to the extent that the laboratory worker found himself with a domestic supply of better glassware, more accurate balances, more highly refractory porcelain, and better reagents than ever before had been at his disposal.

This industry, today really only twenty-five years old, has grown and specialized just as have the industries and sciences which it serves. Certain organizations within the field have developed along specialized lines, while others have acted in the capacity of "service stations" where appliances made by the specialists are centered and distributed. A few combine development, manufacture, and service in one completely integrated unit.

It is natural that an industry in such close contact with science and scientists should profit from that contact, and the same thought methods, the same desire to uncover new facts, and the same critical attitude have prevailed in the laboratories of those making scientific apparatus.

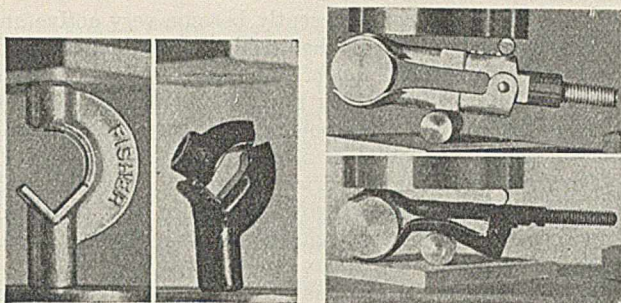


An Explanation of the fourth Table.

- 8 **A** Hook to clear the Grate with.
- 9 **A** Testing Furnace.
- 10 A Pair of Tongs.
- 11 A small Reverberatory, or an open Furnace, for one Retort.
- 12 A Retort.
- 13 Iron Rings to cut Glasses withal.
- 14 A Cone, or Antimonial Horn.
- 15 A Test.
- 16 A Test with its Muffle.
- 17 A Ladle.
- 18 An Ingot.
- 19 A Crucible.
- 20 A left Crucible.
- 21 A Pair of Tongs to take a Pot out of the Fire withal.
- 22 Two Bolt Heads, or Matrasses, made a Circulating Glass of.
- 23 An uncut Body.
- 24 A Body and Head.
- 25 An Egg, or Oval Matrass.
- 26 A Cut Body.
- 27 A Distilling Head.
- 28 A Blind Head.
- 29 A Ballon, or Receiver.
- 30 A Matrass, with its Head.
- 31 A Body, with a double Head.

EARLY CHEMICAL APPARATUS

[As depicted in "A Compleat Course in Chemistry", by Geo. Wilson, Chymist, printed in London in 1709, at the Judges Head in Chancery-lane]



DESTRUCTIVE TESTING TO EVALUATE DESIGN AND MATERIALS

In illustration of the activities of the writer's laboratory, a typical case of development of laboratory apparatus will be described.

Improvement of Laboratory Clamp

It has been wisely said that it is quite as important to know what not to work on as it is to know what to work on. Effort is obviously best directed toward apparatus of broadest application, and a study of the humble but ubiquitous laboratory clamp in all its forms was conducted in order that improvements might be brought about. The common faults of the typical clamp, which had been fabricated in the same way for so many years and yet had exasperated so many users, were brought to light. A survey of users was instituted and the chief faults reported were that they corroded, that the joints froze, that the pressed sheet jaws bent under load, that the screws and wing nuts lacked adequate strength, and that the range of size capacity was too limited.

The problem was then resolved into three phases: a metallurgical problem to be studied with particular attention to corrosion resistance in laboratory atmospheres, a mechanical problem to be approached after a study of the stresses involved in the use of clamps, and a design problem to be worked out after a study of the versatility, positioning, and gripping modes required by the various parts to be clamped.

The metallurgical problem involved corrosion studies of all likely materials, using the ordinary laboratory clamp as control. Platings and finishes were studied in the same way. While no material or combination of materials could be found which was invulnerable to every possible laboratory environment, two were outstanding in comparison with the controls—brass and a strong zinc die-casting alloy. One element of the laboratory environment, mercury, is not usually encountered elsewhere and it was found that a double plating operation was very important to nullify its peculiar activities.

The mechanical problem was attacked through destructive testing of models. Here much was learned which influenced the final designs.

The design problem was naturally conducted concurrently with the mechanical one and the designs most consistent with the requirements of strength, versatility, positioning, and gripping mode were finally evolved.

Early in the course of the work it became evident that the forms would be complex and that they would have to be manufactured either from brass or bronze forgings or from

die castings. Forgings were economically unsuitable and die castings were chosen for those parts which were complex, while brass screw machine parts and tubing were specified wherever possible.

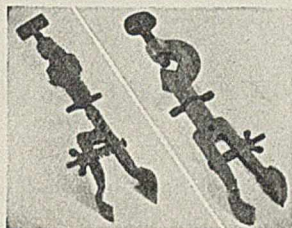
Cooperation with the instrument shop and the die casters finally resulted in samples which were subsequently tested by the development laboratory. Before final approval, samples of all forms were submitted to representative users for trial under field conditions. Based on their suggestions further changes were made and, after final check with those who had presented suggestions, the models were approved and production was begun.

The responsibility of the development laboratory did not end here. Another of its functions is to continue to add new designs to the line, so that over a period of years new clamps and laboratory supports have been added as rapidly as laboratory tests and consultation with typical users have indicated that they will be useful and economical.

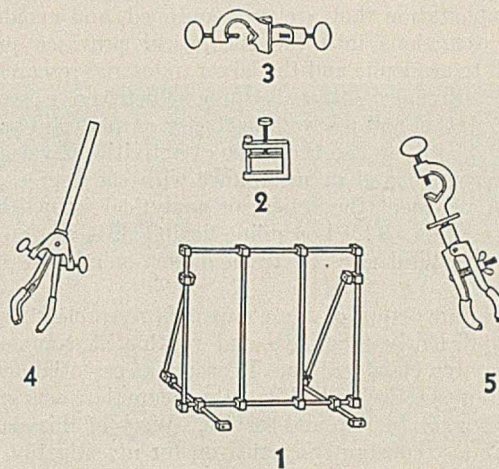
Functions of Development Laboratory

In the course of the development of some types of apparatus a great deal of experience with its uses is necessarily obtained. After an instrument is placed on the market the development laboratory has a further function in working with the potential purchaser in order that he may be able to determine how best to apply it to his needs. Such work has resulted in the development of new techniques and applications and in some cases in published papers.

Because of the breadth of scientific activity and the extent of its specialization, such a laboratory is an interesting and stimulating place. It is necessary to have staff members who are versatile in the extreme, yet who are well acquainted with the important details of many fields of scientific endeavor. This can be done only by careful selection of the personnel, and each individual must have at least one specialty and be able, withal, to work as did the early scientists, as a jack-of-all-trades.



RESULTS OF CORROSION TEST. CONTROL ON LEFT



LABORATORY APPARATUS SUPPORTS

1. Flexible frame support
2. Hose clamp for heavy-walled hose
3. Clamp holder
4. Adjustable clamp with three jaws
5. Clamp with rotatable jaws

The intense specialization which has developed in almost every line of human endeavor and especially in scientific fields, has resulted in special techniques and knowledge in every one of them. Often a method well known to one group of workers would be very useful if applied to the problems of another, yet is entirely unknown to them. The develop-

ment laboratory of the Fisher Scientific Company has taken upon itself the responsibility of serving, in so far as possible, as a sort of multiple liaison officer among the various scientific groups with which it has contact.

During the past decade there has been increasing application of the tools and techniques of radio and television engineering to the problems of the chemist. Recognizing this trend, and aware of the fact that the number of chemists who are adept in the construction of such apparatus is relatively small, the development laboratory has devoted a large part of its attention to apparatus of this nature in an attempt to make these valuable tools readily available. As a result there have already been developed a vacuum-tube voltmeter setup for a variety of titration applications, a photoelectric photometer for quantitative colorimetric analysis, and an instrument for analytical work by means of the dropping mercury electrode.

This line is being followed with diligence and work is now in progress pointing toward the application of electronic and electrical methods to other titration apparatus, to better temperature control of ovens and baths, and even to the determination of the carbon content of steel.

Biological chemistry has recently become very active and the development laboratory has paid a great deal of attention to this particular branch. Likewise, the needs of the hospital laboratory are being studied and present progress indicates that several new tools of value to workers in this field will shortly be available.

The whole line of laboratory apparatus on the shelves of the company is constantly being scrutinized with the intent of making improvements which may result in lower cost and greater utility.

While in the work on clamps cited above the development was initiated entirely within the organization, many others have their beginnings in suggestions of workers in the laboratory. Hence, close contact with the scientific world through reading its literature as well as through attendance at local and national scientific meetings is essential.

It is inspiring to believe that a real contribution to the advancement of science can be made by keeping the scientist supplied with the best possible tools. While it is only too true that "a poor carpenter blames his tools", it is equally true that a good workman produces finer work with finer tools.

Research in Instrumentation

PAUL SHERRICK AND LYNN D. WILSON

E. H. Sargent & Co., 155 East Superior St., Chicago, Ill.

THE research program in the laboratories of E. H. Sargent & Company is restricted as closely as possible to the field of instrumentation. This policy has a multiple objective, in that it maintains intraorganizational efficiency, directs the efforts of the company's personnel into the channels for whose exploitation they are best equipped, and avoids overlapping excursions into the theoretical province properly allocated to academic and the larger industrial research institutions. Obviously, the distinction between research in instrumentation and research in theory or analysis cannot be precisely drawn, and so the scope of activities is varied with each subject in hand in accordance with the degree of adequacy of pertinent theoretical or analytical knowledge initially available. This borderline flexibility is made evident below, in a typical case of extreme distortion into the analytical field.

A large proportion of the company's research work has a nature sufficiently repetitive and routine as commonly to avoid research classification. It occurs as essential activities in invention and design and its data accumulate as a group of facilities for production and service. Work of this classification is almost constantly carried on for investigation of (1) power transmission and conversion, (2) heat generation, transfer, and control, (3) electrical measuring and amplifying circuits, (4) service characteristics of materials and finishes, (5) measurement of physical constants, (6) accuracy limits and requirements of volumetric and gravimetric equipment, etc.

Electrodeposition Studies

Distinct from this function which may be called routine research are the larger and more discrete programs of investigation required for the development or introduction of new analytical instruments of some importance. The company's

last completed program of this character was aimed at an acceleration of deposition rates in electroanalysis. The work on electrodeposition was initiated by a demand for electro-analytical installations expressly suited to high-speed routine analysis of nonferrous metals and alloys. The company was asked to solve a laboratory problem stated approximately as follows:

A group of laboratories analyzing a large number of brass samples a day, and having available in the literature as standardized procedures only the relatively slow-speed methods accepted as quantitative by organizations such as the American Society for Testing Materials, was faced with the economic necessity of reducing the principal time component entailed in the deposition proper. Necessity had forced these laboratories to explore the possible extension of deposition rates and to improvise such equipment and thereby to sacrifice a certain component of mechanical reliability and manipulative efficiency, since practically no instrumental equipment was available to accommodate high current densities. The study of instrumental requirements indicated a necessity for efficient circulation and cooling of the electrolyte, and an elimination of the mechanical hazard of overhead electrode rotating equipment. Furthermore, it was evident that the over-all efficiency of such a laboratory under its normal daily schedule was highly sensitive to the manipulative requirements of apparatus.

A protracted program of design yielded satisfactory solutions of all the principal mechanical difficulties and resulted in sufficient simplicity, flexibility, and durability for the heavy duty to be imposed upon it.

SOLENOID STIRRING. Solenoid stirring has been found distinctly advantageous for high-speed electrodeposition. Its investigation was occasioned by the two principal shortcomings of electrode rotating devices—namely, susceptibility to corrosion from rising acid vapors and delicacy of the alignment requirements when rotating the anode within the cathode. The solenoid method is far from new and was suggested by the publication of Heath (1).

This system was tested by the experimental construction of a great many different solenoids and an optimum result finally secured from a coreless solenoid constructed entirely of nonmagnetic materials and having the following approximate specifications: wire, No. 22 B. & S. gage, enameled copper; turns, 6500; resistance, 150 ohms; winding diameter, 7 inches; core space diameter, 3.375 inches; coil height, 3.5 inches; voltage, 110 direct current; current 0.75 to 1.0 ampere. In the center opening of this solenoid is inserted a close-fitting annular metal water jacket, which in turn surrounds a nonmagnetic stainless-steel beaker well. This cooling arrangement serves the dual function of preventing large temperature rise, resulting from efficiency losses in electrolysis, and at the same time of rapidly dissipating heat produced within the solenoid itself.

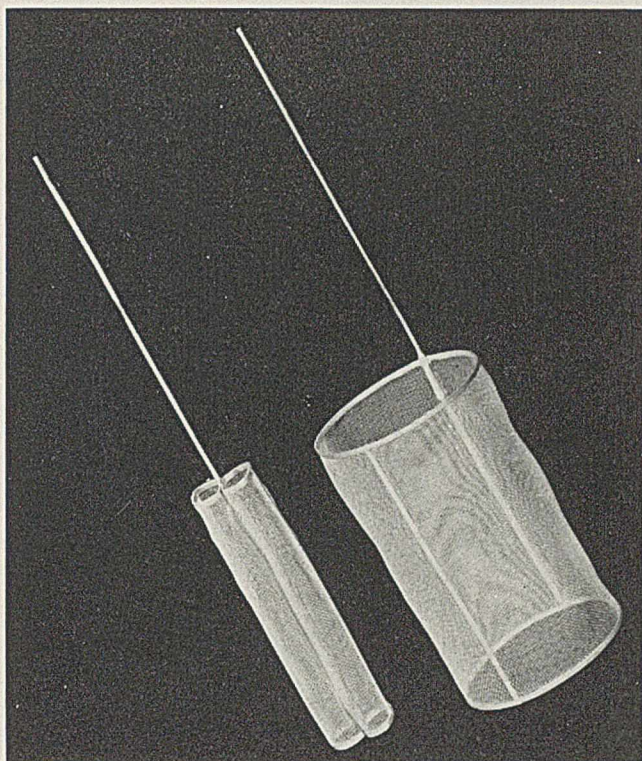


FIGURE 1. ANODE AND CATHODE FOR SOLENOID STIRRING

It is noteworthy that in addition to its structural and manipulative advantages, solenoid stirring is self-adjusting in the sense that increased current densities automatically produce increased rotation rates.

ELECTRODES FOR SOLENOID STIRRING. To employ high current densities large electrode area is essential, since the practical limit for production of good metal coatings is in the vicinity of 0.5 ampere per square inch of effective electrode area, assuming 52-mesh platinum gauze. The same requirement obviously requires a gauze anode. Having determined the maximum practical cathode area, attention was focused on the anode with the aim of bringing its area to a value as nearly as possible equivalent to that of the cathode, and, at the same time of producing a form which would best facilitate thorough mixing under the influence of the motor torque produced by the interaction of magnetic field and electrodepositing current. It was noted that the conventional cylindrical cathode allowed a relatively stagnant condition of the electrolyte confined within it and that diffusion from this quiet area into the rotating stream between cathode and anode was undesirably slow. A special anode form was therefore arrived at by experimentation, consisting of two elliptical tubes united as illustrated in Figure 1.

SIMPLIFICATIONS BY SOLENOID STIRRING. Figure 2 illustrates the possibilities for instrumental simplification as a result of solenoid stirring. Superstructure is eliminated, the solenoids are easily confined beneath a stainless-steel work plate, electrodes may be rapidly manipulated in noncorrosive Bakelite heads operating on grooved rods with centering and depth stops, and control panels are easily condensed to otherwise wasted but readily accessible spaces.

METHODS OF RESEARCH. It was at once evident that while the means of applying high current densities to fast routine electroanalysis had been provided, little was known regarding the practical limits to which such increases of current density could be carried. Furthermore, the ultimate adequacy of the circulatory and cooling provisions could not have been ensured, except as applied under the high current density conditions which were initially assumed to be analytically feasible. It was unavoidable, therefore, to undertake to establish and validate rapid methods for the principal analyses wherever feasible and to establish the maximum current density limits that could be employed. This work was undertaken with full knowledge that it properly belonged in the sphere of the institutional analytical departments, but under pressure of necessity and in the known absence of essential data pertinent to the problem.

The results of this analytical research program comprise 25 electroanalytical procedures shown to be quantitative within the normal requirements for industrial nonferrous control work and offering substantial time-saving in most cases over previous authoritative procedures. It has been found possible, for example, in the deposition of copper from brass to employ a current of 8 amperes throughout most of the deposition, beginning and ending with 5 amperes. The deposit remains coherent and free from burns. In the determination of zinc in brass a current of 6 amperes is allowable and for the same metal in silicon bronzes a current as high as 9 amperes may be used without damage to the deposit. Similar marked increases in current densities with corresponding reductions in deposition time have been effected for many other metal determinations.



FIGURE 2. INSTRUMENTAL SIMPLIFICATION RESULTING FROM SOLENOID STIRRING

The following list of currents as specified in the company's high-speed procedures at present will indicate the saving of time that has been effected in other typical cases.

	Amperes
Copper assay	5 finished at 3
Copper and lead in silicon bronze	5
Copper in nickel-iron alloys	8.5
Nickel in nickel-iron alloys	7
Iron in nickel-iron alloys	4
Zinc	9
Iron in beryllium-copper	4
Nickel in beryllium-copper	7
Copper and lead in steel	5
Aluminum in zinc-base alloys	2
Cadmium in zinc-base alloys	2
Antimony	3
Nickel-cobalt	3
Mercury	3
Silver	3

These procedures have been prepared in mimeographed form for circulation to any laboratories interested in this subject. Formal publication of this work is not contemplated, inasmuch as time and facilities will not permit the collection of a sufficiently great volume of data to determine the ultimate limit of accuracy in these procedures.

Polarographic Research

Current research in the company's laboratories is principally devoted to the establishment of analytical procedures by the polarographic method of J. Heyrovsky. In 1933, when European publications in the field of polarography already totaled well over 200, Professor Heyrovsky proposed that this company undertake to introduce polarographic instrumentation to American laboratories. The study of the subject, begun at that time, revealed its possibilities at a rapidly increasing rate until by 1937 it seemed urgently advisable to make the instrumental facilities and service available at least to those few who were then already engaged in polarographic investigation in this country. As the result of an agreement made with Professor Heyrovsky in that year, the company has established sales and service facilities for this equipment and has presented to all American laboratories the essential data required for its consideration.

It was evident from the beginning that in spite of the extensive literature applying to theoretical and special analytical phases of polarography, a large technical responsibility would be incurred by the company's laboratories, owing to the lack of detailed analytical procedures for the almost unlimited number of analyses for which such procedures can be devised. It was essential in advance of a general sales program to qualify personnel for consultation and methods of research in this field. The situation has been greatly relieved by recent publications in American journals, notably from such investigators as J. Heyrovsky, I. M. Kolthoff, V. W. Meloche, J. J. Lingane, Otto H. Müller, R. H. Müller, J. F. Petras, L. A. Matheson, N. Nichols, G. T. Borchardt, H. Adkins, F. W. Cox, R. M. Burns, O. Kanner, B. Gosman, R. L. Gorman, M. E. Droz, E. S. Peracchio, H. G. Ptering, F. Daniels, E. R. Smith, and C. J. Rodden. References to publications of all these American papers, among others, are compiled in the complete bibliography of polarographic publications maintained by and circulated from the company's laboratories.

At present writing the greatest weight of this current publication is still directed at theoretical considerations and it is therefore necessary in most cases to base recommendations for the use of polarographs on methods of research currently carried out by the company's facilities.

The common question that is asked by prospective polarograph users is simply, "Can we advantageously determine constituents A, B, and C in a substance D?" and in most cases the direct answer to this question can be made most simply by submitting a completed detailed procedure for the desired analysis; because any a priori scheme for such an analysis that might be proposed on the basis of the apparent chemical problems involved requires experimental confirmation of quantitative validity.

This is especially true in polarographic procedures owing to incompleteness of data regarding reduction and oxidation potentials at the mercury electrode under a variety of conditions, to the impossibility of predicting accurately the anodic potentials that will exist under the proposed electrolytic circumstances, to solubility and stability problems in composite

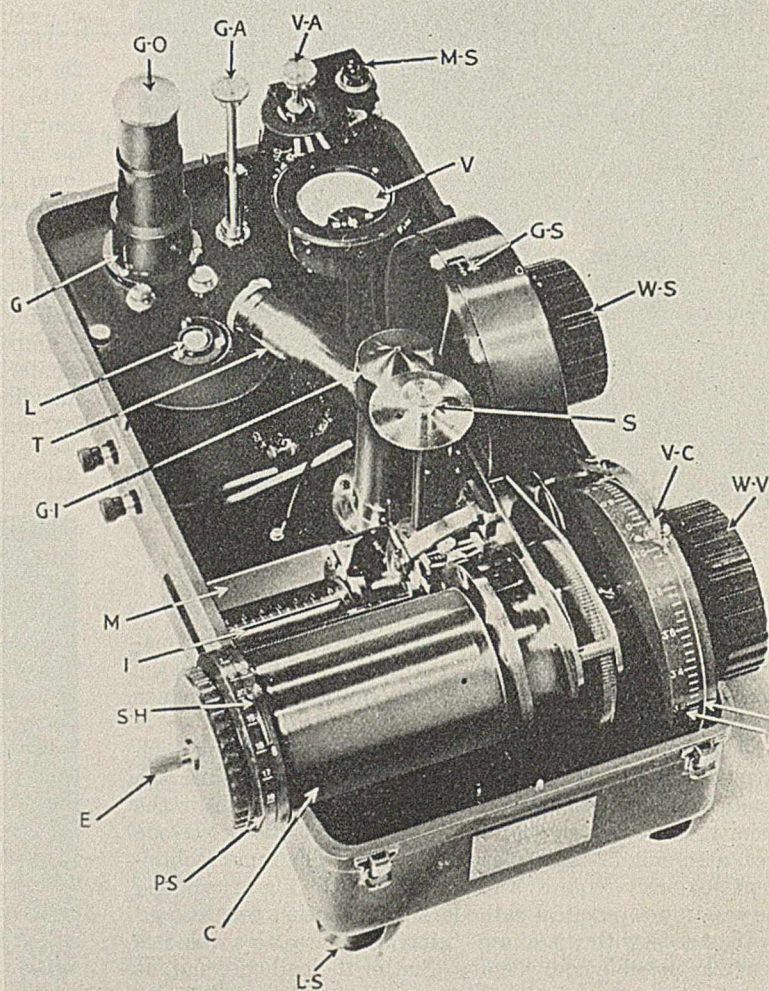


FIGURE 3. INSTRUMENTATION FOR POLAROGRAPHIC ANALYSES

C.	Photographic cylinder	PS.	Displacement scale
E.	Tightening bolt	R.	Slide wire bridge
G.	Galvanometer	S.	Speed adjustment
GA.	Galvanometer arrest	SH.	Shutter control
GI.	Galvanometer light source	T.	Galvanometer telescope
GO.	Zero adjustment	V.	Voltmeter
GS.	Galvanometer shunt	VA.	Voltage adjuster
I.	Exposure slit	VC.	Sliding contact
L.	Level	VS.	Voltage calibration
LS.	Leveling screws	WS.	Shunt knob
M.	Mirror	WV.	Winding knob
MS.	Master switch		

electrolytic solutions not always predictable, to the dependence of the discreteness of closely adjacent current steps upon the existing concentrations of the ions producing them, and, in general, to the novel viewpoint from which polarography requires the analytical chemist to inspect his problem.

DEVisING POLAROGRAPHIC ANALYSES. The peculiarities of the polarographic method are these:

Quantitative measurement is made by the production of a step in a current-voltage curve described under conditions such that the step height is determined by the diffusion rate of the ion being reduced and therefore by the existing concentration of that ion. The dropping mercury cathode employed in a solution containing an excess of other ions less easily reducible than the one momentarily measured provides the above-named condition and further assures a completely polarized cathode, due to which the voltage at which the reduction of a specific ion occurs is related to a thermodynamic property of that ion and is reproducible.

Measurement of the height of a reduction step provides the calculation of an existing concentration of the corresponding substance by comparison of this measurement against that of a step or a step increment produced by a known standard.

Concentration of the substance being determined is unchanged by the analysis.

The determinations of a number of constituents may be made in one polarogram subject to the following general conditions: (1) All substances are in solution in stable ionic states; (2) all substances produce reduction voltages at the mercury cathode within the working range of a 2- or 4-volt bridge; (3) the reduction voltages of all substances are spaced at sufficiently wide intervals; and (4) no substance is in a concentration substantially greater than that of any of the other substances following it in the reduction order. The essential instrumentation is indicated by Figure 3.

This apparently simple set of conditions is found to tax the ingenuity of the analytical chemist attempting to establish a procedure that will effect the greatest possible saving in time in comparison with the more conventional gravimetric or volumetric procedures. In each instance a number of routines are feasible, but almost invariably one of them will eventually be found to require a definite minimum of manipulation and time. Furthermore, the polarographer must always keep in mind the justifications for polarography. If a polarographic procedure is worth devising, it must be faster and less laborious than a corresponding conventional analysis leading to the same approximate accuracy or it must offer an analytical possibility not easily to be secured by the gravimetric, volumetric, or colorimetric procedures. In its microapplications the polarographic method is likely to be found, in many cases, roughly equivalent from the standpoint of time and accuracy to such spectrophotometric methods as may be established for similar work. In such an event the polarographic option may offer the advantages of a lower equipment cost and of a less exacting specialization in technique on the part of the operator.

The most active interest in securing what are sometimes called cookbook procedures in this field is naturally among industrial laboratories where the constant pressure of limited time exists and where new problems are continually challenging the existing knowledge of analysis. Accordingly, the company's present work is planned to produce a group of procedures for certain of the most common routines of industrial laboratories, such as those in metallurgical enterprises. A procedure for complete analysis of zinc-base die-casting alloys, determining copper, lead, cadmium, tin, iron, and aluminum is to be ready for early publication. Results to date indicate the probability of producing all the determinations in either two or three polarograms with a total of no

more than two chemical separations. The possible saving in time that such a method will yield is, of course, evident.

At present the program of research is being confined to problems of rather broad nature and wide interest. In view of the almost unlimited application of the polarograph to the solution of highly specialized problems and the increasing demand for such information, it is hoped that some of this work will be undertaken in the regular course of research at educational institutions.

Literature Cited

- (1) Heath, G. L., *J. IND. ENG. CHEM.*, 3, 74 (1911).

Research on Optical Instruments

C. W. BARTON, *Spencer Lens Company, Buffalo, N. Y.*

EARLY in 1937 the Spencer Lens Company (Scientific Instrument Division of the American Optical Company) found it necessary to expand all facilities to meet a growing demand for optical instruments. A well-organized and competently manned Research and Development Division was the first step in the program and over eighty specialists and technicians have been employed since that time.

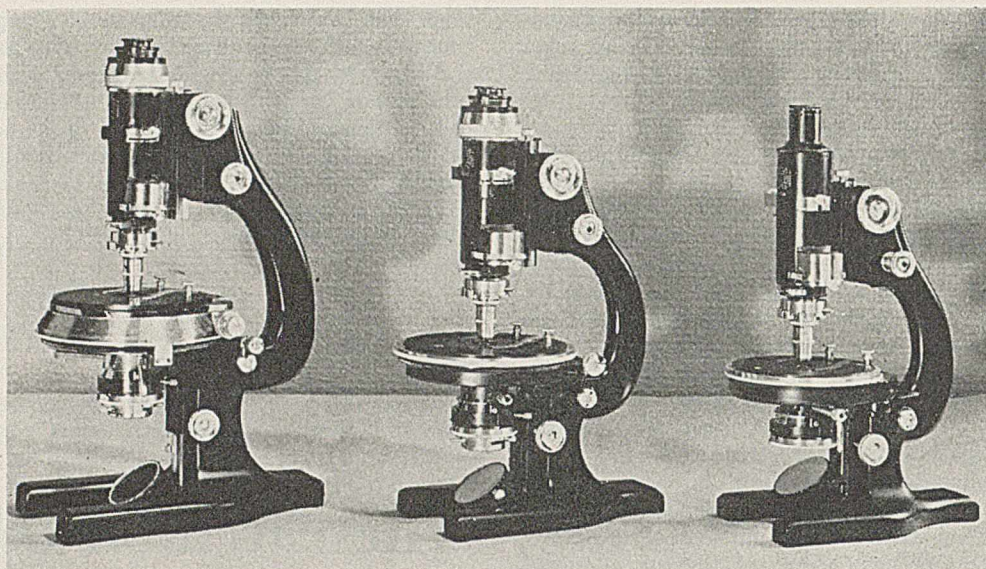
The division has three main functions—research, engineering, and model making—with a personnel to perform, adequately, the specific tasks under these functions. The model-making function is handled in two separate departments, one making the special optical parts and the other the mechanical parts.

A research library, with a trained librarian in charge, ensures availability of reference data and rapid circulation of current scientific and technical literature.

The fundamental research work by members of the Research Department is reported periodically before the scientific and learned societies to which the men belong, and comprises both pure scientific endeavor and research directly related to Spencer products. Contributions to science include lens aberrations, resolution of objectives, growth of organisms, biomathematics, radiometry, color theory, crystal optics, and band spectra. Cooperative research with other laboratories has led to mutual advantage in certain problems.

In addition to the ideas arising from the company's own research work, projects for the Research and Development Division frequently originate in the industrial, educational, research, or governmental laboratories of the country. Scientists or laboratory workers occasionally need equipment which is not commercially available and request assistance from the Spencer Lens Company, either by direct contact or through a Spencer representative in the field. If the needed equipment is a suitable product for the company to manufacture, it may become the subject of a problem, and be assigned to a member of the research staff.

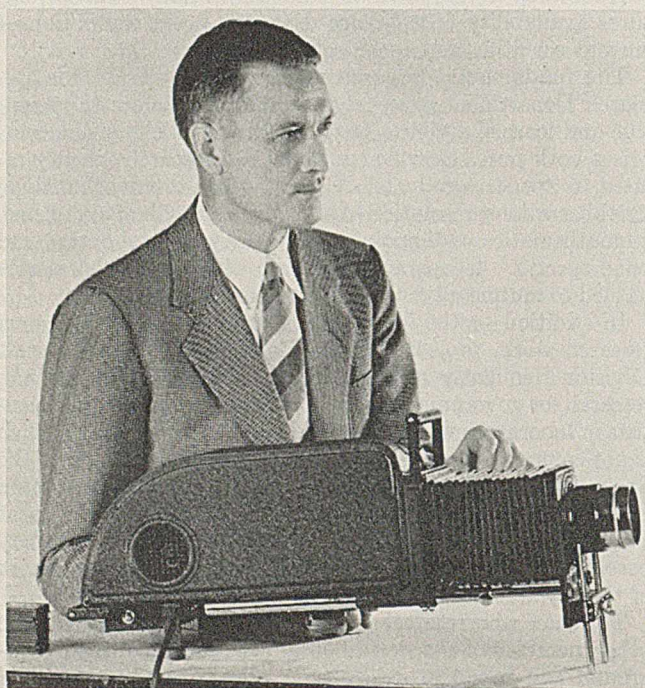
The man who receives this responsibility may consult the other members of the staff to obtain the benefit of their background and broad experience. A group may be formed of several men chosen from the various departments of the Research and Development Division.



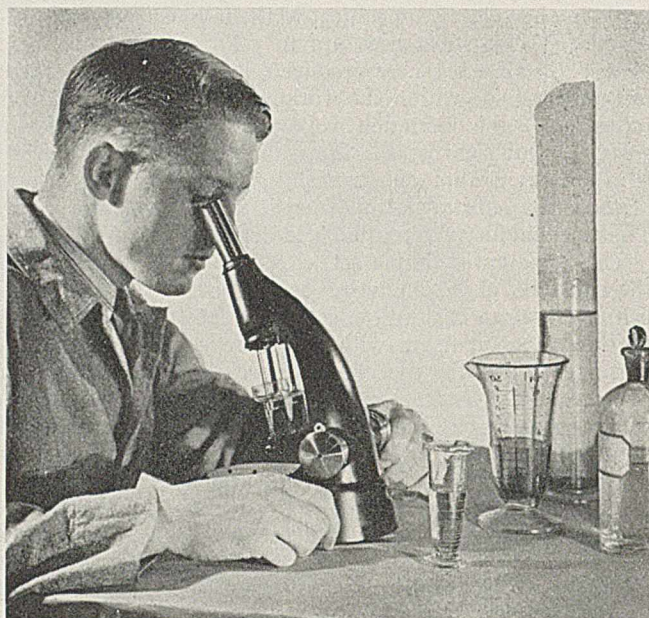
POLARIZING MICROSCOPES

The usual order of work on a project is to survey the field and decide on general principles, necessary precision, and optical and mechanical features. Drawings are then prepared and a pilot model is constructed under the supervision of the Engineering Department and given a rigorous test under operating conditions. This severe, practical testing is to ensure stability, accuracy, convenience, durability, and suitability for the task for which it was designed.

The pilot model may be sent to other laboratories to be certain that it meets the demands for which it was designed. Only after it has been improved as much as possible is it approved by the Sales Division and turned over to the Production Division for manufacture.



SPENCER DELINEASCOPE



DIRECT-RESULT COLORIMETER

In pursuit of their work, the men in the Research Department have greatly simplified optical computation. A careful study of existing colorimeter designs has made possible the development of a simplified optical system for the colorimeter. Improved methods of measuring ventilation and the flow of heat have been devised, reducing temperatures and increasing illumination in projection instruments.

Methods have been developed for testing optical glasses so that their optical properties can be specified within tolerances that will ensure a satisfactory product.

Microtome knives have been studied—sharpening methods, bevels, cutting angles, hardness, and grain structure—in relationship to biological problems. Spectrophotometric data have been applied to the routine quantitative determination of biological materials by colorimetric methods.

The Testing of Chemical Balances

ARCHIBALD CRAIG, Mars, Penna.

VERY little has been written on methods of testing a balance for accuracy. The writer has found only one thorough treatment of the subject (1), written from the manufacturer's point of view.

A perfect balance should have a rigid beam, as light as possible for its capacity, supporting three straight and sharp agate knife edges exactly equidistant from each other, in the same plane, and parallel both horizontally and vertically.

The edges are made by grinding and polishing in two planes, so that the internal angle is a good compromise between sensitivity and durability. Most balances are finished with an internal angle between 90° and 120° . German makers prefer the sharper angle. American practice varies, but the most important makers prefer a more obtuse angle for ordinary analytical balances, and 90° for assay and microbalances.

The edge should be in a single vertical plane, and preferably a straight line. During repairs some edges are finished on a slightly convex plate. If there is equal curvature on both sides, the edge will be in a vertical plane but low in the middle. This gives high sensitivity at first, but the edge rapidly becomes dull.

Makers prefer to get high sensitivity by cutting out some of the middle of the edge rather than departing from the straight line. A convex edge, resting on a short part of the middle, permits the beam or stirrup to twist when free, and is intolerable.

A perfectly sharp edge is an impossibility.

Even when new, an edge should be microscopically rounded, so that it rolls over the plate instead of turning on a line. As a result of the rolling movement of a dull edge, the bearing lines of all edges are shifted, the middle in one direction and the ends in the other, so that the effective beam length on the high side is longer. That increases the speed of the swing, but as the displacement reaches zero when the beam is horizontal, dullness is not in itself a cause of error.

When the rounding is large, the stirrup is thrown off center by the turning of the edge, causing it to tilt, but this movement is opposite to that of the beam, so that the temporary beam error is partly corrected.

A serious result of the rounding of a dull edge is that when weights are displaced to right or left on the pan the tilting of the stirrups may be unequal, and cause a notable shifting of the rest point. Theoretically this should not occur, for there is a joint in the hook of the stirrup which should keep the stirrup plate level even when the pan is aslant. Friction in the joint, however, is often enough to cause tilting and consequent errors in weighing. The only balance which is fully guarded against such tilting is the Austrian Ruprecht, which has knife-edge bearings in the pan supports below the stirrups. It is important with other balances to see that the hook swings freely.

Balances which have grooved stirrups suffer more displacement when dull than those which have flat plates, as the edge tends to climb up the curved face of the stirrup agate and rest on a line farther from the middle of the edge than is possible with a flat plate. Such balances should be avoided except for rough work.

As the edges wear down, the end bearings become lower than the middle, causing a lowering of sensitivity with increased load. When weighings are finished by measuring the deflection of the pointer, there should be no such variation, or else it should be charted for different weights.

The lowering may be expressed as the ratio between (a) a

definite amplitude of swing with empty pans (say, 5 divisions of the scale of an ordinary American balance, or 2.5 divisions displacement of the rest point), and (b) the amplitude with a definite weight added (say, 100 grams).

In ordinary rider balances used to weigh to 0.0001 gram, the ratio should be as much as 5 to 4. A balance used for rough work, particularly for weighing out, where only small weights are used and the counterpoise is adjusted to rapid swing and low sensitivity, is not affected either in speed or accuracy by dullness of the knife edges, if the edges are smooth and not irregularly chipped.

It is possible to set the end edges higher than the middle, so that weight on the pan causes the center of mass to rise and the sensitivity to be greater than when the pans are empty. This is tolerable, except for deflection reading, within a narrow range, say 5 to 5.5, but it is evidence of unskillful work.

If the edges are tilted front and back with respect to each other, the beam length is not directly altered, but there are other bad results. There is an increased lag in the swing. The stirrup tends to shift when freed, increasing the error due to horizontal parallax. The wear on the edges is increased with danger of chipping. The edges should be free from tilt within visible limits.

If the edges are out of parallel horizontally, so that one end of an end edge is farther from the middle edge than the other, the effective beam length will change with every shift in the bearing point of weight on the edge. Such a shift may be caused by slipping of the stirrup front or back, due to a tilted edge or play in the lifting assembly of the stirrup.

Friction in the joints of the stirrup with shifting of the weights on the pan, front and back, is the principal cause of error from this parallax. The Ruprecht balance is well protected against this error by transverse edge bearings below the stirrups, but most other balances are subject to it when old.

The angular variation of the end edges from the middle should be not more than one minute of arc, and preferably a quarter of that.

Tests

The following tests will be useful to show whether the edges of an old balance need sharpening or regulating, and to learn the condition of a recently repaired balance or a new balance of cheaper grade. Some balances on the market are well designed and of good material, but carelessly built. Some of them are very good and are well worth buying if the bad ones can be discovered in time and rejected.

EDGES. If an edge is chipped, it can be seen with a $10\times$ lens, and felt by drawing the edge of a fingernail along it. A few nicks will not affect the use of the balance, if there is still some of the original edge on both sides of its middle. If the bearing is on broken parts there will be an erratic swing and failure to repeat.

A sharp edge may be known by its ability to plane off a real shaving, not merely crumbs, from the thumb nail. A straight edge will make a clean sweep of a film of oil spread on an optical plane surface. A plane piece of glass can be bought from a manufacturing optician. Approximate results can be obtained by taking several small pieces of plate glass, at least 0.3 cm. (0.125 inch) thick and 1.25 cm. (0.5 inch) wide, cleaning them well, and pressing them together two at a time. The interference bands will show which are most nearly flat. If one is found which has straight lines in one direction, so that a straight edge will sweep it, it will do.

After an edge is shown to be straight, it can be used to test the middle bearing plate of the balance. This is particularly necessary in old balances which have two plates on the pillar. They may both be flat but not in the same plane, and if so they will soon spoil a sharp edge.

A straight edge will clean the plate when slanted in either direction, but one that is curved may give two different impressions. A gap in the middle of the edge is a good thing if it is not more than one third of the total length, and if the rest of the edge is in a straight line.

If a balance has edges dull but still able to make a clean sweep of the plate, it should be saved if possible for rough work, as such edges are practically indestructible.

BEAM LENGTH ERROR AND LEVEL OF KNIFE EDGES. Find the rest point with the pans empty. Add 1 mg. and record the deflection. Put 100 grams on each pan, add weight to the original rest point, reverse the weights, adjust again, and record half the algebraic sum of the two corrections. Now add 1 mg. and record the deflection.

The beam length error is measured by the correction made necessary by the added weight. In a good new balance it will be not more than 0.2 mg. per 100 grams. An error of more than 1 mg. should not be tolerated.

If the edges are level with each other, the deflection per milligram will be the same with or without the 100 grams. This will be the case with a good new balance. For an old balance used for fine work it should be not lower than 5 to 4. For a repaired balance it should be at least 5 to 4.5. Ends slightly high, as much as 5 to 5.5, may be tolerated for a rider balance. It will take them longer to get low.

TILTING OF KNIFE EDGES AND VERTICAL PARALLAX. Take a small piece of plate glass, about 0.25 cm. (0.1 inch) thick, 4.25 cm. (1.7 inches) wide, and 15 cm. (6 inches) long, test it with a micrometer to make sure that it is of equal thickness at the corners as evidence that its faces are parallel, and with parallel jaw pliers chip away a rectangular gap in one side of it, 1.25 cm. (0.5 inch) shorter than the distance between the middle and end knife edges. The gap should be deep enough to permit the projections to cover the full length of the two edges without touching the beam or its attachments.

Cut off one end of the strip to leave a projection that will slip through the beam and cover the middle edge with room to turn. Correct the length of the gap at the other end if necessary, so that the other side of the other end will rest freely on the end knife edge.

Get reflected light along the glass, hold the plate firmly on the middle edge, and tilt it until it touches the end. A jeweler's loupe may be used for better vision. If no tilting of the edge is visible, the parallelism is sufficiently good.

The test for tilting can also be made gravimetrically as described below.

HORIZONTAL PARALLAX. Take a smooth wire paper clip, straighten it, and bend it double about 2.5 cm. (1 inch) from one end, so that the two legs almost touch. With the doubled end make a right-angle hook about 5 mm. long. Hang this hook on the lower cross bar of the stirrup if the bar is horizontal. If it is not, fasten a narrow strip of metal across it to make a support for the hook.

Measure down to the regular hook on the stirrup, and make another hook turned up, leaving it just long enough to hold the pan. Hang the pan on this hook, put the stirrup in place, and then bend the hook around until the pan hangs free. Bend the free end of the wire so that it will pass both the regular stirrup hook and the pan bow with the least interference, and bring it straight down so that the whole will hang vertically. Cut another clip for a counterpoise.

Tilt the double hook slightly so that the weight of the pan will rest entirely on the wire from the lower hook. Measure the length of the knife edge and the inside length of the stirrup bar. Open the double hook to a width that will throw the weight of the pan slightly inside the stirrup frame, and under a point just inside the end of the knife edge. Measure the distance between the position of the supporting wire of the hook when at one side

of the stirrup and its position at the other side. In most balances this distance will be from 6 to 7 mm. The distance can be kept constant by setting the hook each time with the free wire against the side of the stirrup.

Set the hook at the back of one stirrup and hang the pan on it. Put the counterpoise wire on the other pan, disregarding a slight difference in weight. Make a weight correction to zero rest point, hang the pan in the front of the stirrup, and correct again. Record the difference in the weights.

Test the sensitivity—that is, the deflection per milligram—of the balance for both positions, if the glass plate test is not used. If the two edges are level with each other the sensitivity will be the same. It should not vary more than 5 to 4.5.

The difference in weight of the two pan positions should be not more than 0.5 mg., though two or three times that will not cause any serious error. It is not uncommon to find repaired balances with an error of 30 mg.

The angle of parallax can be calculated from measurements. For example, on a balance with a 40-gram pan and a 7-cm. beam arm, taking a distance of 7 mm. on the knife edge, 1-mg. variation in the weight of the pan indicates an angle of 51 seconds.

For inspection the weight difference will be sufficient.

Balances of the Sartorius type, which have stirrups pivoted to tilt front and back, need special treatment. Set a small metal plate on the plate holder of the stirrup to lift the pivots slightly and prevent tilting. Tie the parts together, keeping the thread away from the agate plate, take off the loop below, and substitute one made of wire of the same length down to the hook, with two notches 7 mm. apart. The pan can then be hung in two positions, and the parallax determined.

Schulze's method of testing for parallax is to take off the stirrup and hang the pan by a hook on the knife edge. Some balances have enough overhang of the agate to hang a loop of wire on the end of the edge with a hook below. If this cannot be done, a grooved hook can be made to fit over the end of the edge, with another hook below to support the pan under the end.

The writer prefers in all cases to have the stirrup in position, both to protect the edge, and to make sure that the supporting points are in the true edge, and not in an accidental irregularity.

LIFTING MECHANISM. When the beam is lifted, each knife edge should be separated about 0.0125 cm. (0.005 inch) from its plate, with weight on the pans. A slip gage can be used, or a sheet of ordinary note paper. The paper should slide in freely, as most papers are slightly less than the given thickness. After inspecting the middle gap a fair guess may be made at the gaps under the stirrups, which may be hard to reach.

The lift should be vertical. A gap should not show at one end of the knife edge before the other, and neither the pointer nor the stirrups should show any tilting when raised. This is highly important, for a tilt greatly increases the danger of chipping and battering the edge. Too large a gap is bad for the same reason. The given gap measurement is a maximum, to make sure that there will always be some gap. If there is no gap, the edge will wear against the plate from the vibration of the building.

The stirrups should be perfectly centered above the knife edges, so that they will have no sidewise tilt when let down.

A sidewise kick of the pointer when lifted is evidence that the stirrup lifts are either unequal in height or not vertical.

PAN RESTS. Pan rests which work by gravity need little attention except to see that they move freely and hold the pans in the proper position.

Pan rests which are brought by levers into fixed positions should not be set too high, as they will cause the stirrup to tilt and may damage the edges.

Acknowledgment

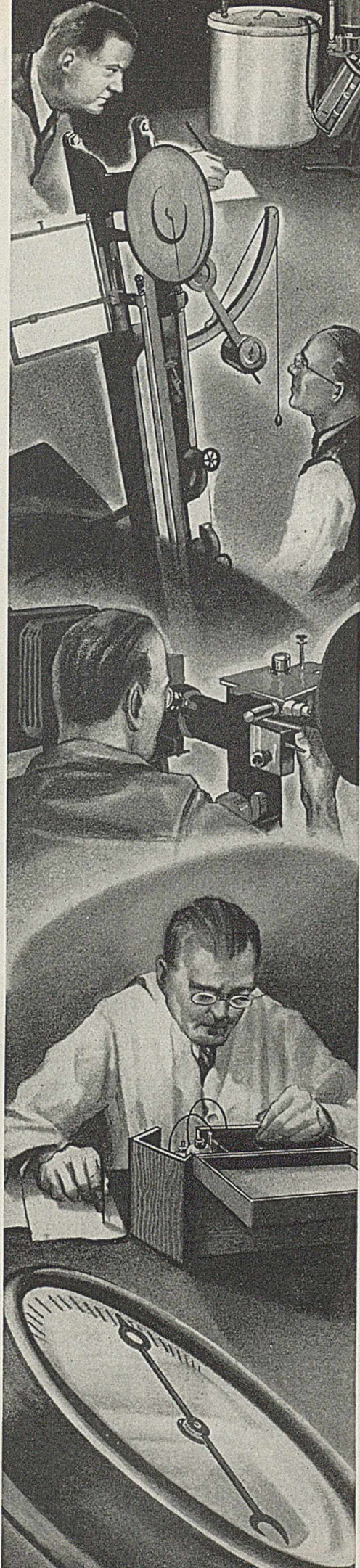
Acknowledgment is due to A. T. Pienkowsky of the National Bureau of Standards for advice and assistance in preparing these tests.

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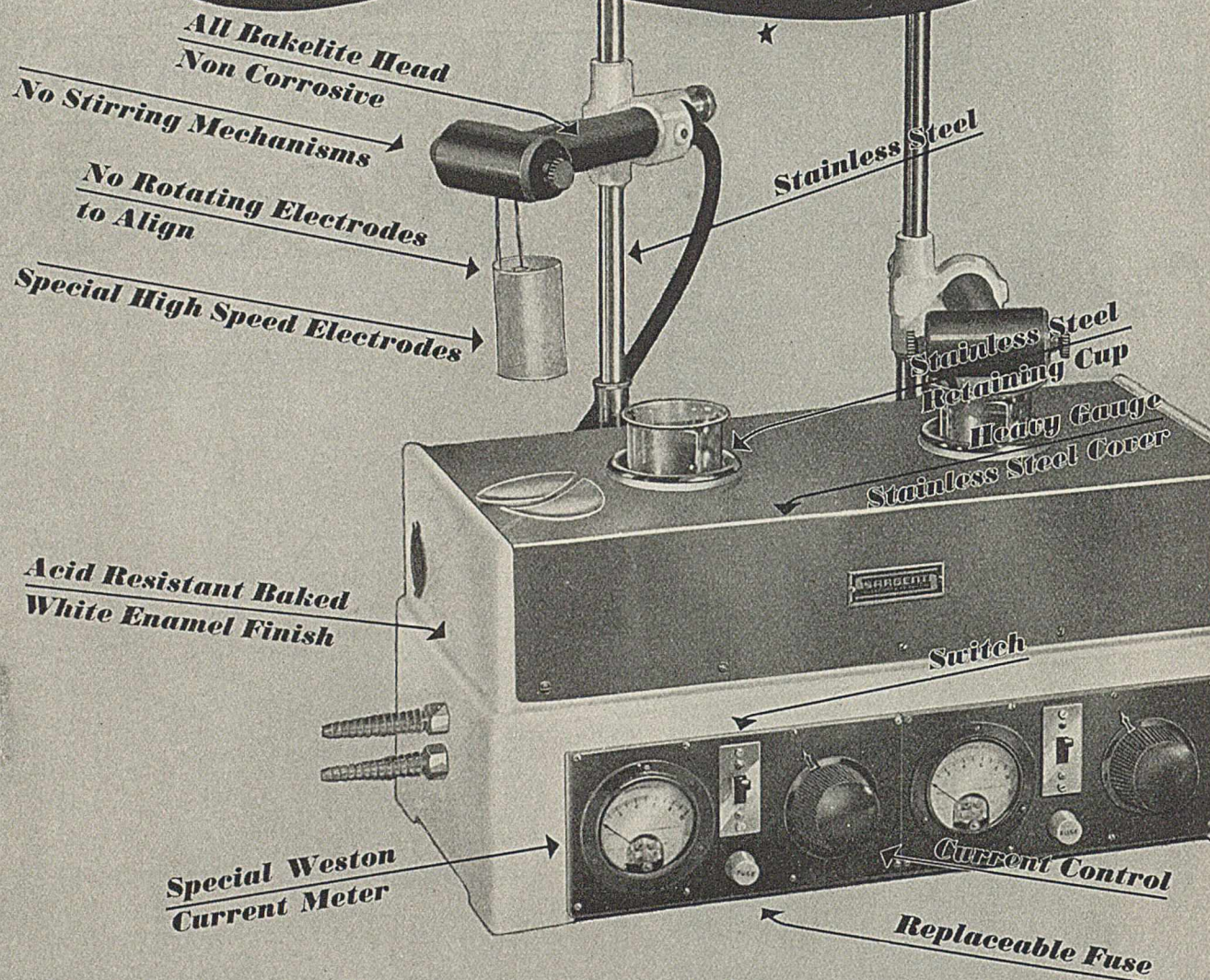


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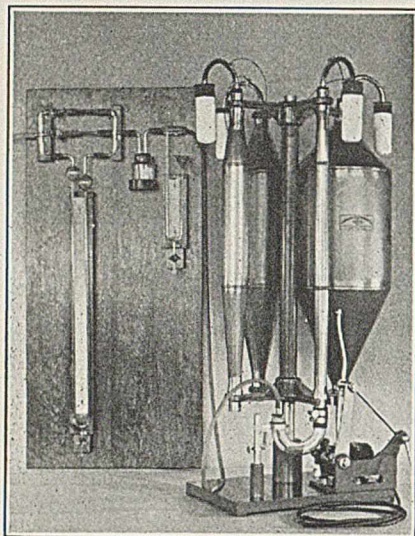
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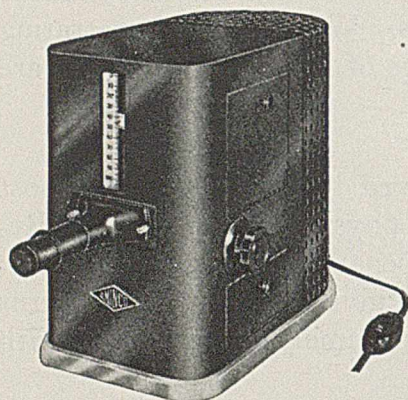
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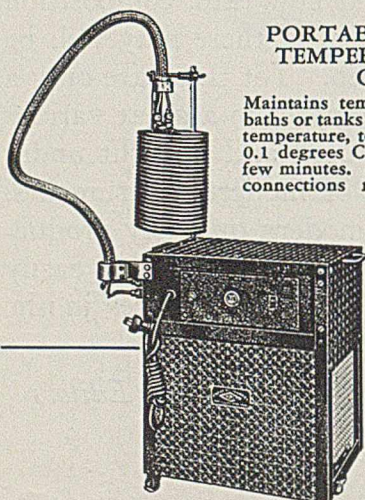
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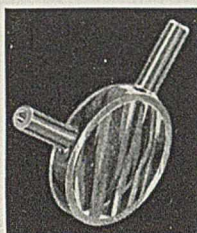
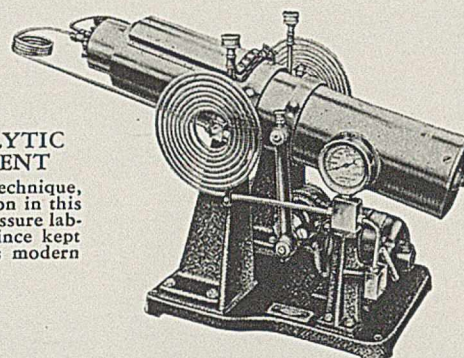
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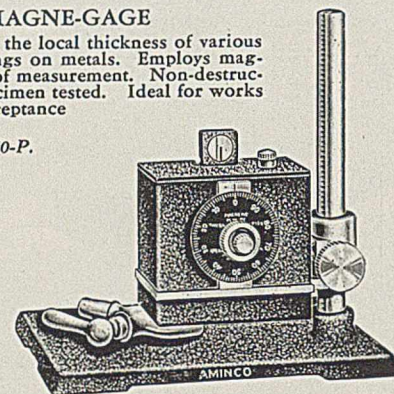
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Frequent cross references have been made to eliminate duplication of firm listings. For example—BENDING TESTING, See Tensile Testing, shows that firms which supply the former are identical with the firms supplying the latter. This does not imply that the equipment is identical.

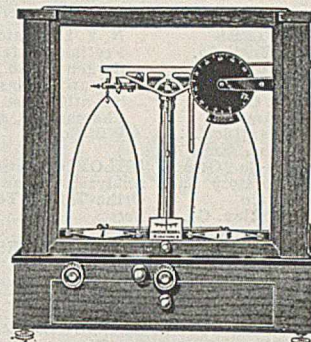
The more important laboratory instruments have been separately indexed as warranted by their importance. For example POTENTIOMETERS are separately listed and have not been cross-indexed to ELECTRICAL MEASURING.

Proprietary trade names have been used only where the trade has no other aptly descriptive designation for the device.

If the product or name for which you are searching is not found in the Index, please communicate with the Advertising Office, Industrial and Engineering Chemistry, 332 West 42nd St., New York.

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For complete name and address of companies listed in this index consult pages 30 and 32.



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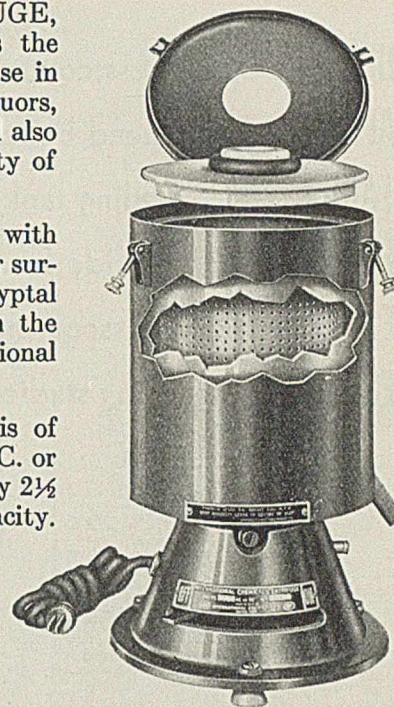
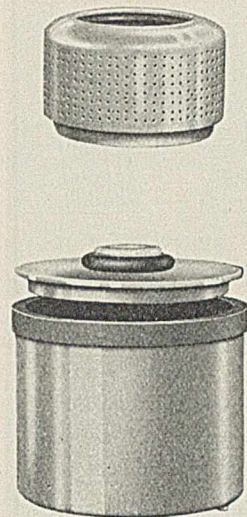
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Thomas Co., A. H.
Will Corp.

G

GAGE TESTERS
American Inst. Co.
Amthor Test. Inst.
Crosby Steam G. & V.
Holz, H. A.
Manning, M. & M.,
Olsen Test. Mach.
Refinery Sup. Co.

GAGES, Liquid Level, See Liquid Level

GAGES, Mercury
Amthor Test. Inst.
Bacharach Ind. Inst.
Bailey Meter
Braun Corp.
Builders Iron Fdy.
Central Scientific Co.
Connelly Iron S.
Daigger & Co., A.
Defender Auto. Reg.
Dubrovin, John
Eimer & Amend

GAGES, Micrometer, See Lineal Dimensions

GAGES, Paint Film, See Paint Testing

GAGES, Precision, Measuring, See Lineal Dimensions

GAGES, Pressure, See Pressure and Vacuum

GAGES, Strain, See Tensile Testing

GAGES, Thickness, See Lineal Dimensions

GAGES, TORQUE
Riehle Div.
Saxl Inst.
Toledo Scale

GAGES, McLeod, See McLeod

GAGES, Vacuum, See Pressure and Vacuum

GAGES, Wire Measuring, See Lineal and Dimensions

GAS ANALYZERS
Bacharach & Co.
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Bushnell & Nevius
Cambridge Inst. Co.
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Everson Mfg.
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Gow-Mac Inst. Co.
Greiner, Inc., Emil
Greiner & Co., Otto
Hays Corp.
Leeds & Northrup
Linde Air
Mine Safety
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Permutit Co.
Precision Scien. Co.
Precision T. & I.
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Scientific Glass App.
Scientific Inst. Co.
Tagliabue Mfg.
Taylor Inst. Cos.
Thomas Co., A. H.
Uehling Inst. Co.
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Willson Products

GAS DENSITY METERS, See Gas Analyzers; Gravimeters

GAS LEAK INDICATORS
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GAS METERS, See Flowmeters

GASOMETERS, See Gas Analyzers

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Eimer & Amend
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Greiner & Co., Otto
Hellige, Inc.
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La Motte Chem. Prod.
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Sargent & Co., E. H.
Scientific Glass App.
Taylor & Co., W. A.
Thomas Co., A. H.
Thwing-Albert
Will Corp.

EMULSION AND DEMULSIBILITY

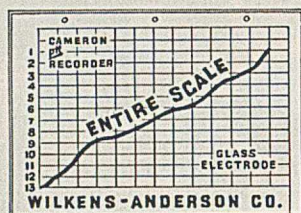
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Daigger & Co., A.
Eimer & Amend
Fisher Scientific Co.
Greiner, Inc., Emil
Greiner & Co., Otto
Int'l. Equip. Co.
N. Y. Laboratory Sup.
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EXPANSION, See Dilatometers

EXPOSURE, See Illumination Measuring

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GRATINGS, Diffraction, See Spectrometers

GRAVIMETERS, See Specific Gravity; Gravimeters

GRAVITOMETERS
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GRAVITY INDICATORS AND RECORDERS, See Specific Gravity

GREASE TESTING
See Oil Testing

GROUND RESISTANCE, See Electric Measuring

GUMMED TAPE TESTING, See Paper Testing

H

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Scientific Glass App.
Shore Inst. & Mfg.
Suter, A.
Test. Mach. Inc.
Thomas Co., A. H.
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HELIOSTATS, See Meteorological Measuring

HUMIDISTATS, See Humidity

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Taylor Inst. Cos.
Thomas Co., A. H.
Will Corp.

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Eimer & Amend
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Fisher Scientific Co.
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Greiner & Co., Otto
Hellige, Inc.
Indust. Insts. Inc.
La Motte Chem.
Leeds & Northrup
Nat'l. Tech. Labs.
N. Y. Laboratory Sup.
Sargent & Co., E. H.
Scientific Glass App.
Taylor & Co., W. A.
Thomas Co., A. H.
Thwing-Albert
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N. Y. Laboratory Sup.
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HYGROTHERMOGRAPHS, See Humidity

I

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IMPACT TESTING, See Tensile Testing

INDUCTANCE, See Electrical Measuring

INSULATION TESTING, See Electrical Measuring

INSULATION, Compactness
Central Scientific Co.

J

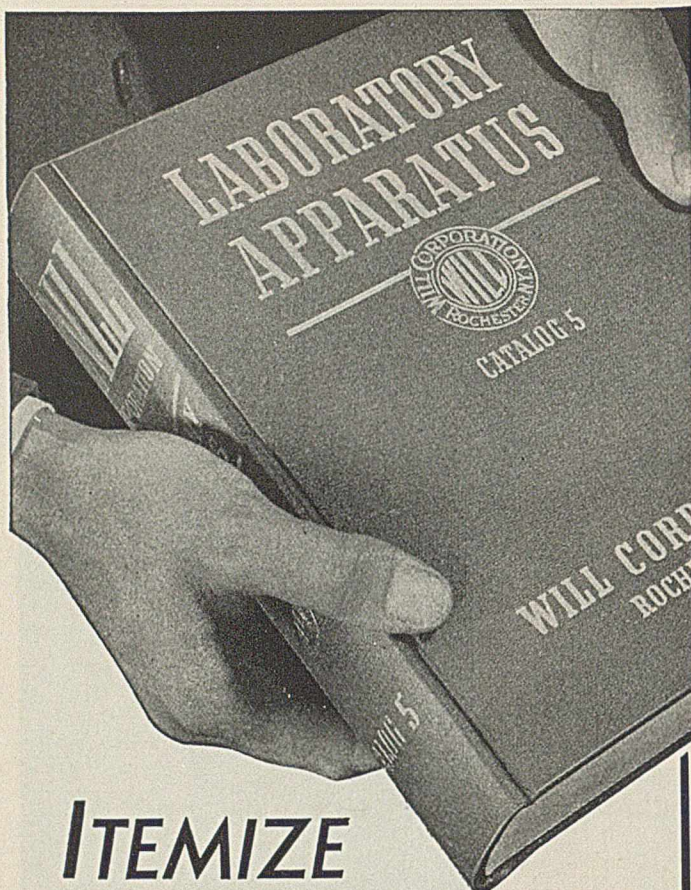
JELLY STRENGTH
See Glue Testing

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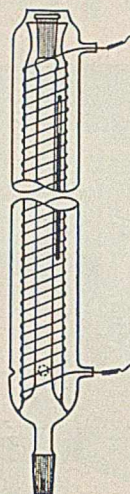
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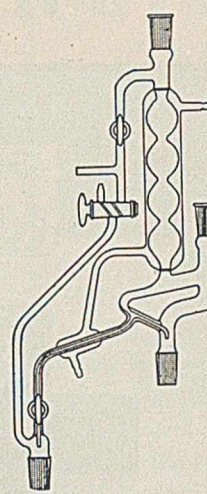
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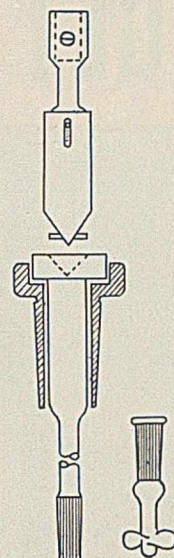
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Precision T. & I.
Spencer Lens Co.
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LIGHT METERS, See Illumination

LINEAL DIMENSIONS

See also Optical Flats
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Scherr Co., George
Scientific Glass App.
Sheffield Gage
Starrett
Thomas Co., A. H.
Will Corp.
Zeiss, Inc., C.

LIQUID LEVEL

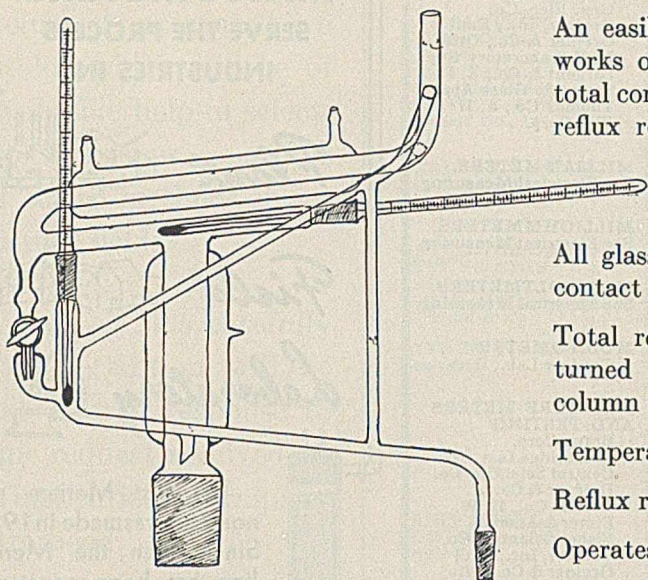
Arca Regulators
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LIQUID METERS, See Flowmeters

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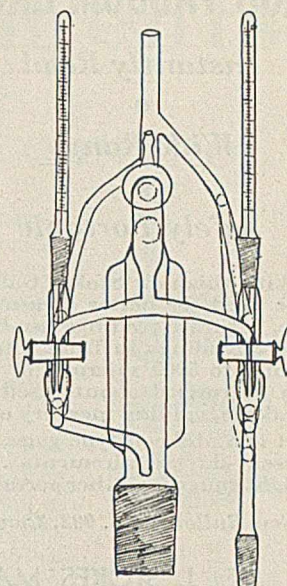
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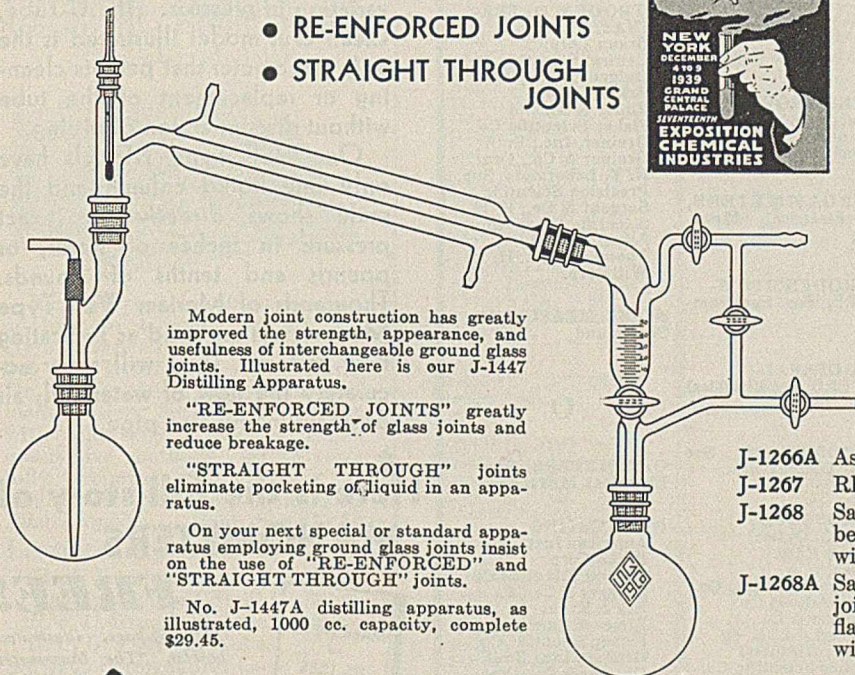
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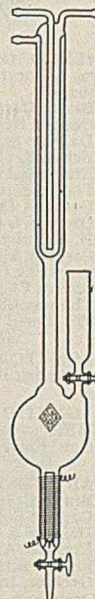
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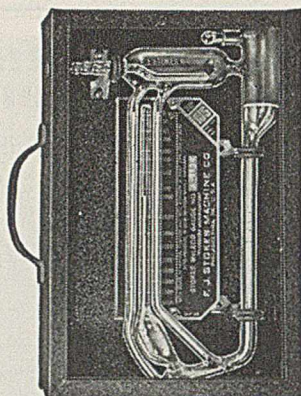
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M

MCLEOD GAGES, See
also Laboratory Supply
Houses

Braun Corp.
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See Electrical Measuring

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CHINES, Lineal, See
Lineal Dimensions

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See Electrical Measuring

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See Electrical Meas-
uring

MICRODENSITOM-
ETERS, See Spectrom-
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MICROFARAD-
METERS, See Electrical
Measuring

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Lineal Dimensions

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Sargent & Co., E. H.
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Spencer Lens Co.
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Will Corp.
Zeiss, Inc., Carl

MICROVOLT METERS
See Electrical Measuring

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Greiner & Co., Otto
N. Y. Laboratory Sup.
Sargent & Co., E. H.
Scientific Glass App.
Thomas Co., A. H.
Will Corp.

MILLIAMMETERS,
See Electrical Measuring

MILLIOHMMETERS,
See Electrical Measuring

MILLIVOLT METERS,
See Electrical Measuring

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AND TESTING

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Eimer & Amend
Fisher Scientific Co.
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Greiner & Co., Otto
Industrial Inst., Inc.
N. Y. Laboratory Sup.
Precision Scientific
Sargent & Co., E. H.
Scientific Glass App.
Scientific Inst. Co.
Tagliabue Mfg.
Thomas Co., A. H.
Will Corp.

N

NEPHELOMETERS,
See Color Measuring

NIGROMETERS, See
Color Measuring

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MINATION

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Daigger & Co., A.
Eck & Krebs
Eimer & Amend
Fisher Scientific Co.
Greiner, Inc., Emil
Greiner & Co., Otto
N. Y. Laboratory Sup.
Precision Scientific
Sargent & Co., E. H.
Scientific Glass App.
Scientific Inst.
Thomas Co., A. H.
Will Corp.

NOISE MEASURING
See Sound

O

OHHMMETERS, See
Electrical Measuring

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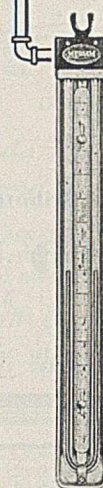
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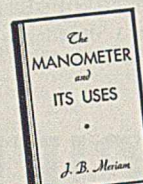
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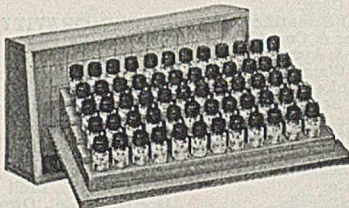
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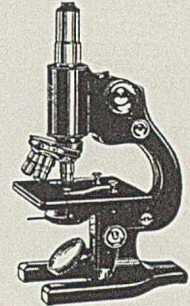
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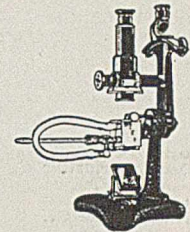
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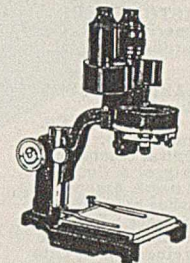
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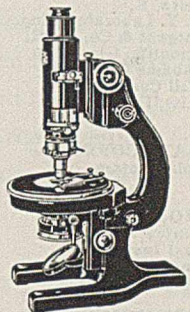
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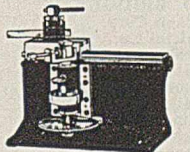
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& Recording,** See Hy-
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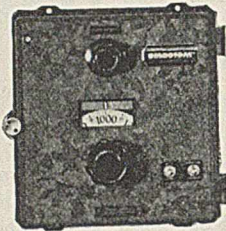
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Stillman Co.
Tagliabue Mfg.
Thomas Co., A. H.
Will Corp.

STRAIN TESTING (Except Glass), See Ten- sile Testing

STRESS TESTING, See Tensile Testing

STRETCH (Except Metals)

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Suter, A.
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Greiner & Co., Otto
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Permutit Co.
Sargent & Co., E. H.
Scientific Glass App.
Tagliabue Mfg.
Thomas Co., A. H.
Will Corp.

SULFUR DIOXIDE

See Gas Analyzers

T

TACHOGRAPHS, See Tachometers

TACHOMETERS

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Boulin Inst. Corp.
Braun Corp.
Bristol Co.
Brown Inst.
Central Scientific Co.
Daigger & Co., A.
Eimer & Amend
Electric Tach. Co.
Electric Speed Ind.
Esterline-Angus
Fisher Scientific Co.
Foxboro Co.
Greiner, Inc., Emil
Greiner & Co., Otto
Jones-Motrola
Manning, M. & M.
N. Y. Laboratory Sup.
Precision T. & I.
Sargent & Co., E. H.
Scherer Co., G.
Scientific Glass App.
Sticht & Co.
Thomas Co., A. H.
Westinghouse E. & M.
Weston Elec. Inst.
Will Corp.

TAPES, See Lineal Dimensions

TELEMETERING

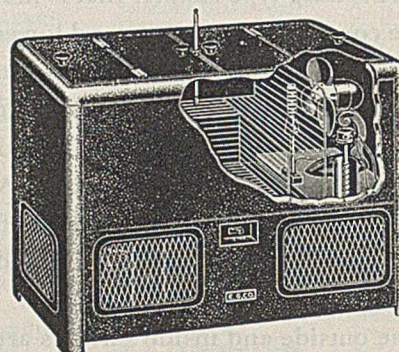
American Meter Co.
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Bailey Meter
Barber-Coleman
Bristol Co.
Brown Inst.
Electric Valve
Esterline-Angus
Foxboro Co.
King-Seely Corp.
Manning, M. & M.
Mason Neilan
Republic Flow
Richardson Scale
Spence Eng. Co.
Tagliabue Mfg.
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Westinghouse E. & M.

TELESCOPES

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Perkin, E. & M.
Spencer Lens Co.

TEMPERATURE, See also Pyrometers; Ther- mometers; Thermopiles; etc.

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Foxboro Co.
Friez & Sons
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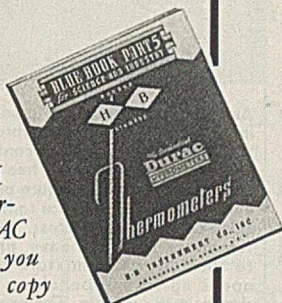
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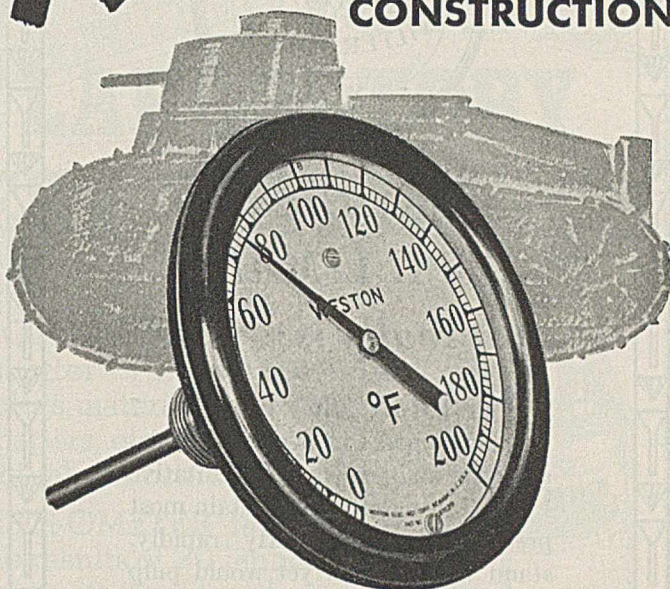
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Saxl Inst. Co.
Scientific Glass App.
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Will Corp.

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Sargent & Co., E. H.
Scientific Glass App.
Thomas Co., A. H.
Will Corp.

THERMOCOUPLES
See Pyrometers

THERMO-HYDROMETERS, See Hydrometers

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Gotham Inst. Co.
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Industrial Inst. Co.
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Liquidometer Corp.
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Marsh Corp., J. P.
Mason Neilan Reg.
Moeller Inst. Co.
N. Y. Laboratory Sup.
Palmer Co.
Partlow Corp.
Pecorella Mfg. Co.
Permutit Co.
Phila. Therm. Co.
Powers Reg. Co.
Precision T. & I.
Rep. Flow Meters
Sarco Co., Inc.
Sargent & Co., E. H.
Scientific Glass App.
Scientific Inst. Co.
Tagliabue Mfg.

TEST WEIGHTS, See Weights

TESTERS AND TESTING MACHINES, See subject of measurement

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Greiner & Co., Otto
Gurley, W. & L. E.
N. Y. Laboratory Sup.
Perkins & Son
Sargent & Co., E. H.
Saxl Inst. Co.
Scientific Glass App.
Sheffield Gage
Spencer Lens Co.
Suter, A.
Taber Inst. Co.
Thomas Co., A. H.
Thwing-Albert Inst.
Will Corp.

THERMO-AMMETERS
See Electrical Measuring

THERMO-ANEMOMETER
Willson Products, Inc.

TENSILE TESTING

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Eimer & Amend
Ferner Co.
Fisher Scientific Co.
Gen. Elec. Co.
Greiner, Inc., Emil
Greiner & Co., Otto
Holz, H. A.
Linde Air Prod.
N. Y. Laboratory Sup.
Olsen Test. Mach.
Perkins & Son
Riehle Test Div.
Sargent & Co., E. H.
Saxl Inst. Co.
Scientific Glass App.
Scott, H. L.
Suter, A.
Test. Mach. Inc.
Thomas Co., A. H.
Thwing-Albert
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Thwing-Albert Inst.
Weston Elec. Inst.
Will Corp. | TIME MEASURING,
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also Stop Watches
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Auto. Temp. Con.
Barber-Coleman
Braun Corp.
Bristol Co.
Brown Inst.
Calculagraph Co.
Cambridge Inst. Co.
Central Scientific Co.
Clebar Watch Co.
Coleman Elec. Co.
Cramer Co., Inc.
Daigger & Co., A.
Dunn-Struthers
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Frober-Paybor
Gaertner Scientific
Gordon Co.
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Greiner & Co., Otto
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Inter. Mktng. Corp.
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Walser Auto. Timer
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Wheelco Inst. Co.
Will Corp. | TOTALIZERS, Fluid
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Eppley Lab., Inc. | THERMOSTATS
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Friez & Sons
Fulton Syphon
General Elec.
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Greiner & Co., Otto
H-B Inst. Co.
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Partlow Corp
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Powers Reg. Co.
Precision Scientific
Precision T. & I.
Robertshaw Therm.
Sargent & Co., E. H.
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Westinghouse E. & M. |
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TERS, See Manome-
ters |
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Leod type, See McLeod |
| | | VACUUM GAGE, Pirani
Type; See also Pressure
and Vacuum
Distillation Prod. Inc. |
| THREAD TESTING
See Textile Testing | TORSION, See Tensile
Testing | VACUUM MEASURING
See Pressure and Vacu-
um; Barometers; Gages
Mercury and McLeod;
Manometers |

VALVE POSITION INDICATORS

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Builders Iron
Manning, M. & M.
Taylor Inst. Cos.
Tefft-Jackson

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Cargille, R. P.
Thwing-Albert Inst.

VARNISH TESTING
See Paint Testing

VENTURI METERS,
See Flowmeters

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Electrical Res. Prod.
Gen. Elec. Co.
RCA Mfg. Co.
Sheffield Gage

VISCOSIMETERS

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Gardner Lab., H. A.
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Greiner, Inc., Emil
Greiner & Co., Otto
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Analysis Sets

WATER METERS, See
Flowmeters

WATTHOUR METERS
See Electrical Measuring

WATTMETERS, See
Electrical Measuring

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Atlas Electric Dev.

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Exact Wt. Scale
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Greiner, Inc., Emil
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Jarrell-Ash Co.
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N. Y. Laboratory Sup.
Precision Scientific
Sargent & Co., E. H.
Scientific Glass App.
Seederer-Kohlbusch
Thomas Co., A. H.
Torsion Balance
Troemner, Henry
Volland & Sons
Will Corp.

WEIR METERS, See
Flowmeters

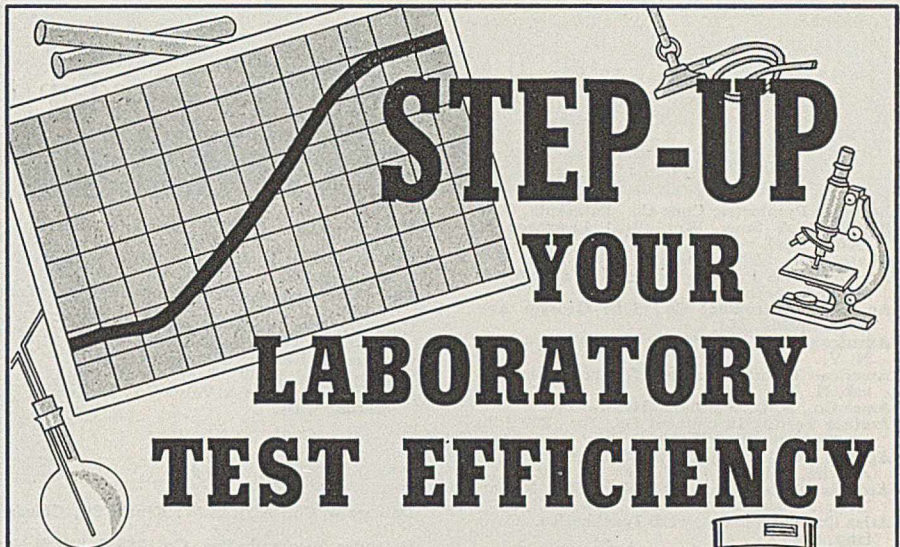
X

X-RADIATION MEASURING, Diffraction
Gaertner Scientific
Jarrell-Ash Co.
G. E. X-Ray Cory.

X-RAY SPECTROMETERS, See Spectrometers

Y

YARN TESTING, See
Textile Testing



STEP-UP YOUR LABORATORY TEST EFFICIENCY

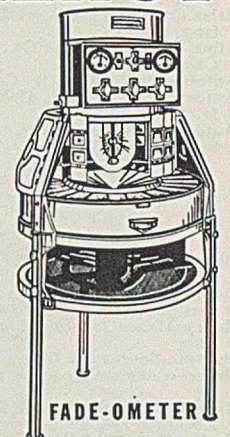
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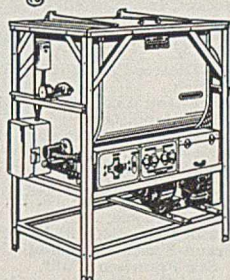
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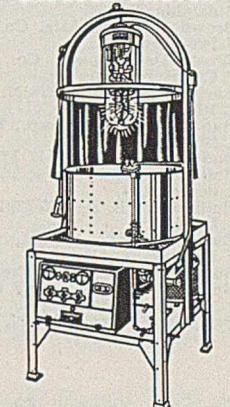
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B

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C

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 Cash Co., A. W., Eldorado at Third St., Decatur, Ill.
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 Chicago Apparatus Co., 1735 N. Ashland Ave., Chicago, Ill.
 Clafin Co., G. L., 150 Dorrance St., Providence, R. I.
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 Colloid Equipment Co., 50 Church St., New York, N. Y.
 Connelly Iron Sponge & Governor Co., 3154 S. California Ave., Chicago, Ill.
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D

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 Dubrovin, John, 2947 N. Kenneth Ave., Chicago, Ill.
 DuMont Labs., Inc., Allen B., 2 Main St., Passaic, N. J.
 Dunn, Inc., Struthers, 150 N. Juniper St., Philadelphia, Pa.

E

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 Eastern Engineering Co., 45 Fox St., New Haven, Conn.
 Eberbach & Sons, 200 E. Liberty St., Ann Arbor, Mich.
 Eck & Krebs, 131 West 24th St., New York, N. Y.
 Eclipse Fuel Engineering Co., Rockford, Ill.
 Edison Electric Controls Div., Thos. A. Edison, Inc., 51 Lakeside Ave., West Orange, N. J.
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 Electronic Research Lab., Niles Center, Ill.
 Elgin Softener Corp., Elgin, Ill.
 Empire Laboratory Supply Co., 559 W. 132nd St., New York, N. Y.
 Engelhard, Inc., Charles, 90 Chestnut St., Newark, N. J.
 Eppley Laboratory, Inc., Newport, R. I.
 Ess Instrument Co., 30 Irving Place, New York, N. Y.
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 Everson Mfg. Co., 210 W. Huron St., Chicago, Ill.
 Exact Weight Scale Co., West Fifth St., Columbus, Ohio

F

Fairbanks, Morse & Co., 600 S. Michigan Ave., Chicago, Ill.
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 Fee & Stempel, Inc., 4949 N. Pulaski Rd., Chicago, Ill.
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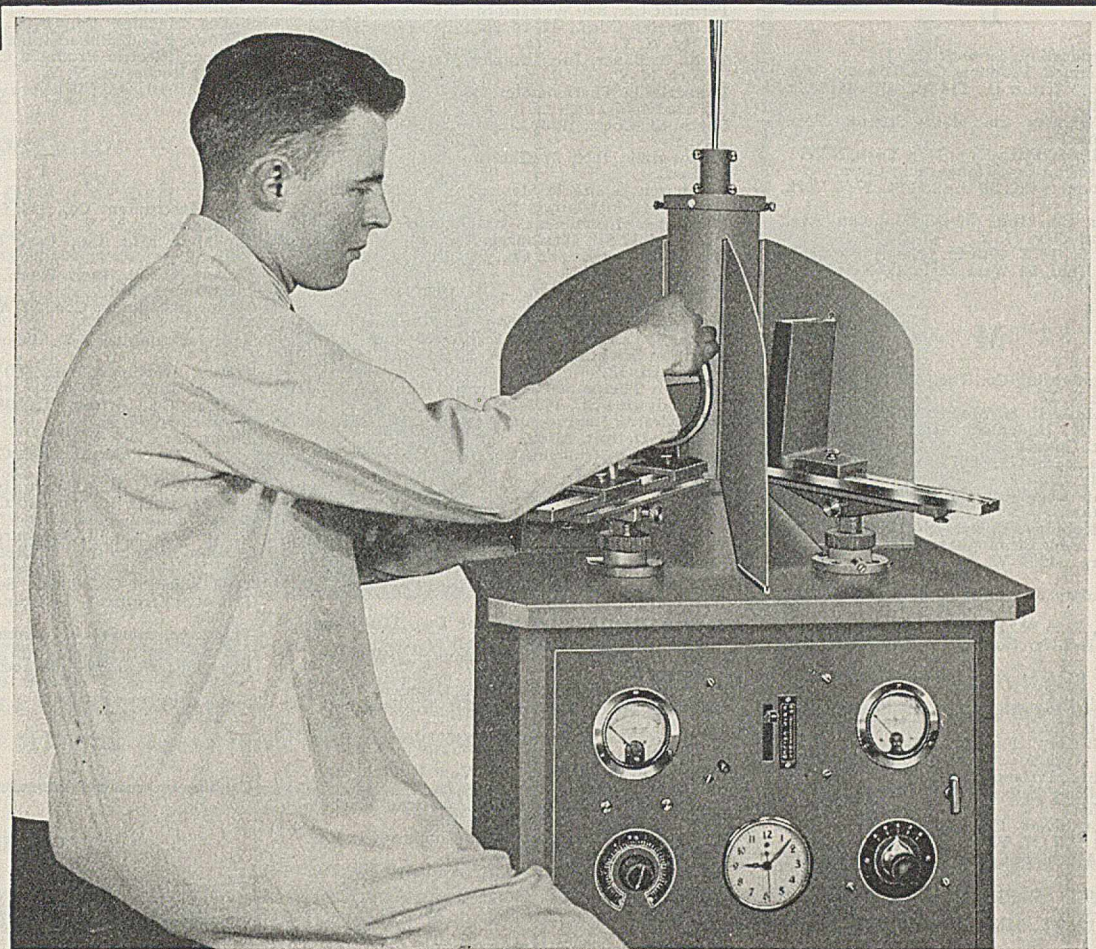
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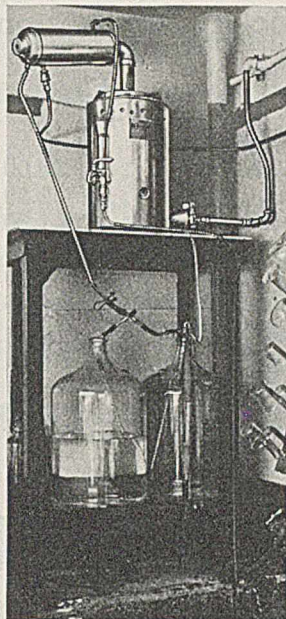
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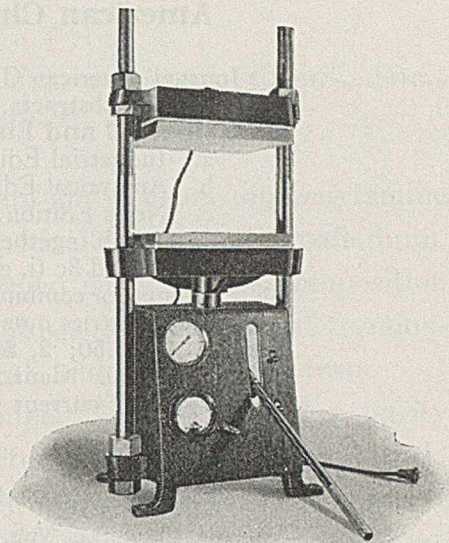
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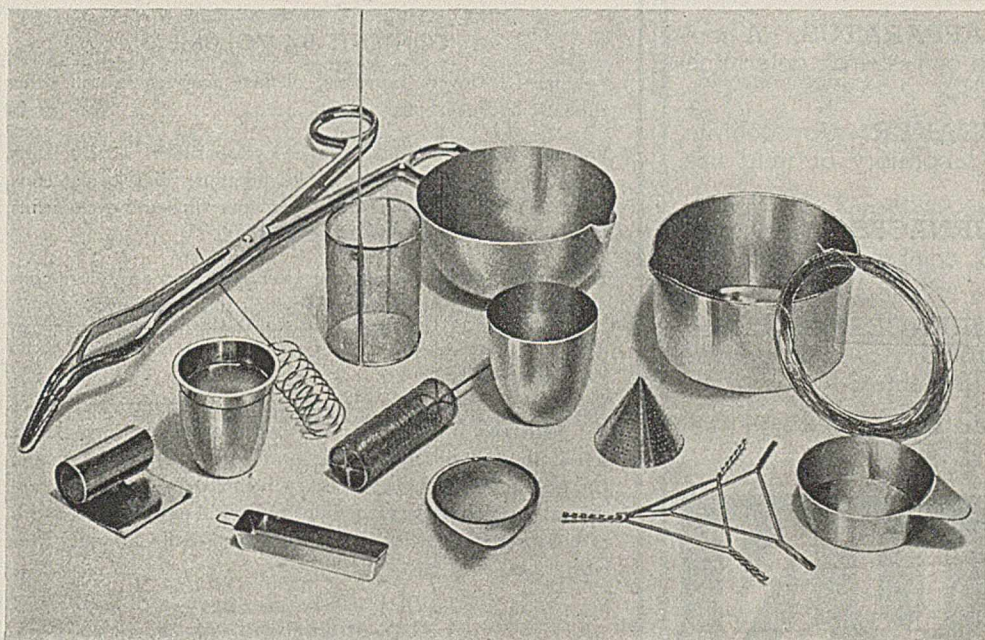
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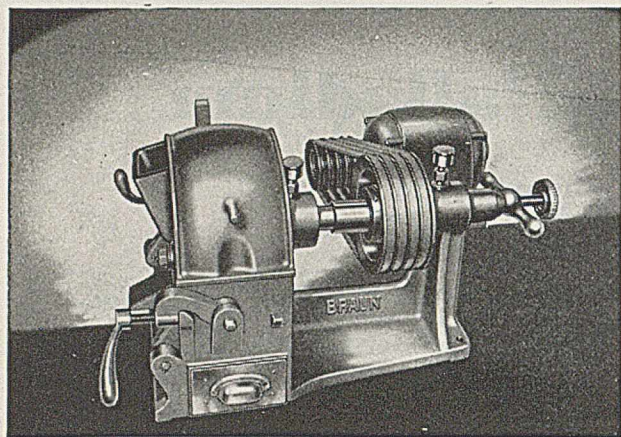
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