

NOTES

Composition of Coconut Shells

BY REGINALD CHILD AND S. RAMANATHAN

L. C. Fleck and co-workers recently have reported the results of standard analytical determinations carried out on coconut shells.¹ We have had in hand since 1935 a study of the development of the coconut fruit involving the analysis of husks, shells and endosperm of fruits at various stages of maturity.² Since this study will not be complete for some time, we are placing on record the results of our determinations on mature coconut shells, which are in general agreement with those of L. C. Fleck and his colleagues, except for the somewhat higher figure for cellulose determined by a different method.

The only figures on record before those referred to above seem to be those of A. L. Winton,³ who in 1901 applied the usual methods of Food and Drug analysis to the detection of powdered coconut shells in spices, which adulteration he stated to be a very common practice in the United States. His figures are not otherwise of particular interest. A figure of 56.19% was recorded for "crude fibre," but A. G. Norman⁴ has emphasized the unreliability of this determination, since the "crude fibre" fraction obtained bears no definite relationship to the structural constituents of the original material.

J. B. Boswell⁵ has investigated the composition of certain nut-shells, and has reported that of the seed coat of Brazil nuts (*Bertholletia excelsa*). In this material lignin appears to be present in larger and cellulose in lesser proportion than in coconut shells. It is of interest, however, to note that the cellulose in Brazil nut shells was found to be associated largely with xylan, and we have been able by suitable hydrolysis to isolate crude xylose in considerable amount from coconut shells, and from the "cellulose" fraction thereof. R. W. Tromp de Haas and B. Tollens⁶ obtained "a satisfactory yield" of crystalline xylose by

hydrolysis of coconut shells with 4% sulfuric acid, and stated that this appeared to be the only product of the hydrolysis. The residue gave dextrose on hydrolysis with stronger acid.

We propose to investigate further the xylan fraction of coconut shells and the nature of its association with cellulose.

Experimental

The shells were ground to pass a 100-mesh sieve and the determinations tabulated below were carried out. Pentosans were estimated by the method of A. W. Schorger,⁷ cellulose by the method of A. G. Norman and S. H. Jenkins⁸ and lignin by a modification of the method of H. Schwalbe,⁹ whereby 2 g. of material was treated with a mixture of 10 cc. of 18% hydrochloric acid and 50 cc. of 72% sulfuric acid, the mixture kept cool for two hours and then boiled for thirty minutes after dilution with 300 cc. of water.

TABLE I
COMPOSITION OF COCONUT SHELLS

	On air-dried material, %	On dry weight (at 105°) %	Figures of Fleck, <i>et al.</i> , ¹ based on weight of oven-dry (105°) shells, %
Moisture	8.78	...	6.07
Cold water soluble	0.19	0.20	1.43
Hot water soluble	3.25	3.57	2.67
Alcohol soluble	2.29	2.51	...
Benzene soluble	0.25	0.27	0.19 (Ether soluble)
1% alkali soluble	17.15	18.80	20.53
Ash	0.56	0.61	0.23
Lignin	33.30	36.51	33.30
Cellulose	48.40	53.06	44.98
Total pentosans	26.70	29.27	30.28
Pentosans in cellulose	...	20.54	17.67
Pentosans in cellulose (on basis of cellulose)	...	38.69	39.30

¹ Fleck and others also record % methoxyl, "Hydrolysis no." (loss in cellulose due to 15% H₂SO₄ hydrolysis), % cellulose stable to 15% H₂SO₄ hydrolysis, acetic acid by hydrolysis, and loss in weight of shells due to 2.5% H₂SO₄ hydrolysis from acetic acid determination.

The hot water extract contained 0.160% K₂O calculated as a percentage of the original shells. Potash is the major constituent of the ash.

(7) As adopted by the Cellulose Division of the American Chemical Society; see *Ind. Eng. Chem.*, **15**, 748 (1923).

(8) A. G. Norman and S. H. Jenkins, *Biochem. J.*, **27**, 818 (1933).

(9) H. Schwalbe, *Papier-Fabr.*, **23**, 174 (1925).

(1) L. C. Fleck, W. G. van Beckum, and Geo. J. Ritter, *THIS JOURNAL*, **50**, 2279 (1937).

(2) See "Report and Accounts of the Coconut Research Scheme for 1936" (Ceylon Government Sessional Paper X, 1937, page 5).

(3) A. L. Winton, *Am. J. Pharm.*, **73**, 552 (1901).

(4) A. G. Norman, *J. Agr. Sci.*, **25**, 529 (1935).

(5) J. B. Boswell, *Biochem. J.*, **30**, 971 (1936).

(6) R. W. Tromp de Haas and B. Tollens, *Ann.*, **286**, 303 (1895).

Summary.—The results of standard analytical determinations on coconut shells agree well with those recorded by Fleck and colleagues except for the higher value found for cellulose.

It is proposed to investigate further the nature of the association of xylan and cellulose in coconut shells.

COCONUT RESEARCH SCHEME OF CEYLON
LUNUWILA, CEYLON RECEIVED JANUARY 4, 1938

The Preparation of Diacetone Sugars

BY H. VAN GRUNENBERG, C. BREDT AND WERNER FREUDENBERG

The use of concentrated sulfuric acid as catalyst and dehydrant for acetonizations of carbohydrates has found general acceptance. Several disadvantages of this method are well known; while high (4–5%) concentration of sulfuric acid¹ brings about rapid condensation of acetone, less catalyst leaves considerable quantities of sugar undissolved and favors formation of monoacetone derivatives instead of diacetone sugars. Furthermore, the neutralization of sulfuric acid with anhydrous solid carbonate is time consuming. Other catalysts, zinc chloride,² copper sulfate³ or phosphoric anhydride,⁴ among others, have been suggested; much lower yields however have been obtained as compared to the sulfuric acid method.

In studying the acetonization of *l*-sorbitose we have obtained by using zinc chloride in combination with a mixture of ortho- and metaphosphoric acids very satisfactory results and have developed the method given below which seems to be generally applicable to the preparation of diacetone sugars and superior to older methods in yield, speed and simplicity. The application of this process to the commercial preparation of diacetonesorbitose (ascorbic acid synthesis) has been considered.

The following yields of diacetone sugars were obtained: *l*-sorbitose 85%, *d*-arabinose 90%, *d*-galactose 78%, *d*-mannose 92% and *d*-glucose 75%. In all cases the products isolated were practically pure as checked by analysis and rotation. The yields are based on crystallized or distilled products. Due possibly to the acidic acetonization medium the same diacetone products are obtained which are formed by the use of sulfuric acid as catalyst.

(1) Bell, *J. Chem. Soc.*, 1874 (1935); Ohle, *Ber.*, **71**, 562 (1938); Reichstein and Grüssner, *Helv. Chim. Acta*, **17**, 311 (1934).

(2) Fisher, *Ber.*, **60**, 485 (1927).

(3) Ohle and Koller, *ibid.*, **57**, 1566 (1924).

(4) Smith and Lindberg, *ibid.*, **64**, 505 (1931).

Procedure.—One hundred grams of the very finely powdered sugar is suspended in two liters of dry acetone to which is added in rapid succession 120 g. of fused zinc chloride (sticks) and a homogeneous mixture of 20 g. of phosphorus pentoxide and 40 g. of phosphoric acid (85%). The whole is shaken mechanically until solution is complete, which lasts about two hours. Then, in order to bring the reaction to completion the solution is allowed to stand overnight at room temperature. The mixture is then made alkaline by adding an aqueous suspension of sodium carbonate, the precipitate of zinc carbonate is filtered, washed with acetone, and the combined filtrate and washings are distilled *in vacuo* until most of the acetone is removed.

The resulting aqueous residue is extracted with three 150-cc. portions of benzene or ether, which, after removal of the solvent *in vacuo*, leaves the diacetone product in practically pure form.

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Thermal Data on Organic Compounds. XVIII. The Heat Capacity and Entropy of *t*-Butylethylene

BY WM. D. KENNEDY, C. HOWARD SHOMATE AND
GEORGE S. PARKS

The combination of constancy of freezing point and constancy of boiling point serves as an excellent criterion of the purity of a chemical compound. This criterion has been used frequently by Kistiakowsky¹ and his collaborators in their extremely accurate determinations of the heats of organic reactions. Recently in the case of *t*-butylethylene,^{1b} however, they failed to find a constant freezing point by their methods, although otherwise they had every reason to believe in the extreme purity of the material; and accordingly they invited us to study the fusion behavior of a sample of their material by our calorimetric method.

We have now measured the heat capacities of this olefin between 80 and 298°K. From the temperature change during the fusion of the crys-

(1) (a) Kistiakowsky, Ruhoff, Smith and Vaughan, *THIS JOURNAL*, **58**, 137, 146 (1936); (b) Dolliver, Gresham, Kistiakowsky and Vaughan, *ibid.*, **59**, 831 (1937); (c) Dolliver, Gresham, Kistiakowsky, Smith and Vaughan, *ibid.*, **60**, 440 (1938).

talline material we have estimated the purity of the sample as, at least, 99.8 mole per cent. The heat of fusion, however, is unusually small for such a type of compound and this fact readily accounts for the difficulty encountered by the Harvard investigators in their freezing point tests.

Furthermore, it should be noted that this compound exists in two crystalline forms at low temperatures with a transition point at 124.9°K. In magnitude the heat of transition (12.35 cal. per g.) is almost four times that of the heat of fusion of the Crystals II.

Experimental Results

In principle, the method of Nernst was employed with an aneroid calorimeter in determining the "true" specific heats and the transition and fusion data. The apparatus and details of experimental procedure have been fully described in other places.² In view of the accuracy of the various measurements involved, the error in the experimental values obtained here is probably less than 0.7% at all temperatures.

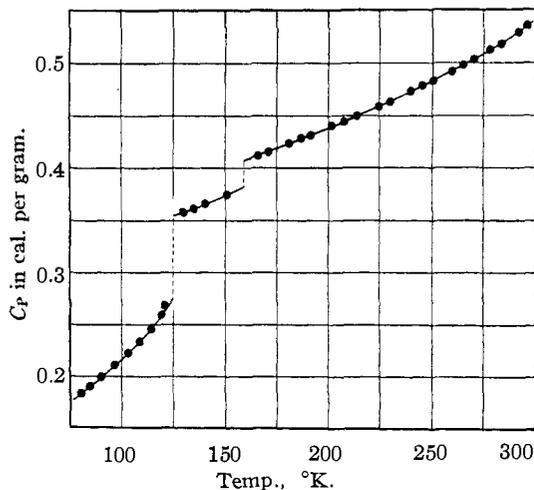


Fig. 1.—The specific heat curve for *t*-butylethylene.

These heat capacity data, expressed in terms of the defined calorie (equivalent to 4.1833 international joules) and with all weights reduced to a vacuum basis, appear in Tables I and II. The specific heat values are also represented graphically in Fig. 1, in which it is noteworthy that pre-melting and other effects of impurities are practically negligible.

Entropy Data

Using the heat capacity data contained in the

(2) Parks, *THIS JOURNAL*, **47**, 338 (1925); also Parks and Kelley, *J. Phys. Chem.*, **30**, 47 (1926).

<i>T</i> , °K.	<i>C_p</i>	<i>T</i> , °K.	<i>C_p</i>	<i>T</i> , °K.	<i>C_p</i>
Crystals I					
80.8	0.1828	97.2	0.2107	113.6	0.2458
85.6	.1903	102.8	.2221	118.8	.2595
90.9	.1990	108.4	.2333	121.1	.2689
Crystals II					
129.5	0.3569	140.2	0.3655	151.3	0.3750
134.4	0.3608				
Liquid					
165.9	0.4104	213.6	0.4492	265.3	0.4972
171.2	.4138	224.7	.4581	270.8	.5027
181.2	.4226	230.3	.4624	277.8	.5119
186.8	.4275	240.4	.4719	283.4	.5182
191.7	.4294	246.0	.4780	291.4	.5287
201.5	.4397	251.3	.4817	295.9	.5348
207.3	.4445	260.2	.4912		

Process	<i>T</i> , °K.	ΔH determination (cal. per g.)		
		I	II	Mean
Transition	124.9	12.34	12.35	12.35
Fusion	158.4	3.11	3.11	3.11

preceding section in conjunction with the third law of thermodynamics, we have calculated the entropy of liquid *t*-butylethylene at 298.1°K. The detailed entropy increments are given in Table III. In this calculation we have employed the extrapolation method (with "class I" constants) of Kelley, Parks and Huffman³ for estimating the entropy increase for the low temperature crystals from 0 to 80°K. The various increments from 80 to 298.1°K. were obtained by the usual methods directly from the experimental data. The result for the molal entropy at 298.1°K. is thus found to be 61.3 cal. per degree. This

Crystals I (0–80°) extrapolated	11.39
Crystals I (80–124.9°) graphical	8.26
Transition $\left(\frac{12.35 \times 84.092}{124.9}\right)$	8.32
Crystals II (124.9–158.4°) graphical	7.32
Fusion $\left(\frac{3.11 \times 84.092}{158.4}\right)$	1.65
Liquid (158.4–298.1°) graphical	24.35
S_{298}° for the liquid state in cal. per degree	61.3

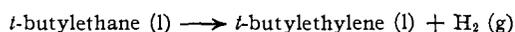
(3) Kelley, Parks and Huffman, *ibid.*, **33**, 1802 (1929). As recently indicated in the paper of Parks, Shomate, Kennedy and Crawford [*J. Chem. Phys.*, **5**, 359 (1937)], the "class II" constants, derived originally for cyclic compounds, may yet prove more accurate in the extrapolation of the entropies of all hydrocarbons, both aliphatic and cyclic. Until this point is definitely proved, however, it seems to us preferable to continue to employ the class I constants for aliphatic hydrocarbons, as in the past.

value is probably reliable to within 1.0 e. u. in an absolute sense and to 0.5 e. u. for comparative purposes.

The molal entropy of the corresponding paraffin, 2,2-dimethylbutane, has been reported recently by Stull⁴ as 64.4 (± 1.8) e. u. Thus the paraffin-olefin difference here is apparently 3.1 e. u., which is a somewhat greater effect than that (2.2 e. u.) found by Parks, Todd and Shomate⁵ in the case of 2,4,4-trimethylpentane and 2,4,4-trimethylpentene-1.

Free Energy of Dehydrogenation of 2,2-Dimethylbutane

The data are now available for the estimation of the free energy change in the dehydrogenation process



For the gas-phase reaction Kistiakowsky and co-workers^{1b} have found $\Delta H_{355} = 30,340$ (± 60) cal. Reducing this value by 250 cal. for the temperature change from 355 to 298°K. and assuming that the heats of vaporization of the paraffin and olefin are practically the same, we now find $\Delta H_{298} = 30,090$ (± 200) cal. In the preceding section we found the entropy difference between the liquid paraffin and olefin to be 3.1 e. u. and in view of the similarities in the extrapolations we now estimate the probable error in this figure as under 1.5 e. u. Accordingly, taking 31.23 e. u.⁶ for the molal entropy of hydrogen, we have $\Delta S_{298} = 28.1$ (± 1.5) e. u. Hence, $\Delta F_{298}^{\circ} = 30,090 - (298.1)(28.1) = 21,700$ (± 500) cal. for the process in question.

This result is in good agreement with the figure 21,000 (± 400) cal. reported by Parks, Todd and Shomate⁵ for several other cases involving the dehydrogenation of paraffins to yield monosubstituted ethylenes.

(4) Stull, *THIS JOURNAL*, **59**, 2727 (1937).

(5) Parks, Todd and Shomate, *ibid.*, **58**, 2505 (1936).

(6) Giaque, *ibid.*, **52**, 4825 (1930).

DEPARTMENT OF CHEMISTRY
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A Semi-Micro Sublimation Apparatus

BY C. M. MARBERG¹

In the course of some work on the isolation and identification of lipoids undertaken in this Laboratory, it became necessary to submit certain frac-

(1) The Otho S. A. Sprague Memorial Institute and the Department of Pathology, The University of Chicago.

tions to high-vacuum sublimation. The theory, technique, and several designs of apparatus for this procedure are contained in an article by Hickman and Sandford.² The apparatus of Werner,³ which utilizes a ground joint and a water-cooled condenser is useful only with minute quantities of material, which are condensed on a cover glass. When used with larger quantities of material, omitting the cover-glass, the sublimate may become contaminated with grease upon withdrawal of the condenser, due to accidentally touching the ground joint. Finally, when sealed to the high-vacuum line, it is necessary to break the apparatus loose after each time it is used. Carothers and Hill⁴ devised a molecular still which overcame the possibility of contamination of the distillate with grease, by using a large, flat-ground joint. Their apparatus was improved by Strain and Allen⁵ who simplified the heating arrangement, sealed the water-cooled condenser into the upper dome, and added a ground joint by means of which the apparatus could be attached to the high-vacuum line. Since we desired to cool our condenser below temperatures possible with water or circulating fluid, none of this apparatus, which utilizes water-cooled condensers, was suitable for our work.

The apparatus devised for our work,⁶ shown in the accompanying diagram, has the advantages that (1) low temperatures may be used for the condensing surface, since the condenser may be cooled with carbon dioxide, liquid ammonia, or liquid air, (2) the ground joints, including the stopcock, are all interchangeable, and (3) the section carrying the stopcock can be sealed to the high-vacuum line (we use a mercury pump backed by a Cenco Hyvac) and may be left in place, since it does not come into contact with either the crude material or the sublimate.

A solution of the crude substance is placed in the unattached lower vessel, the design of which may be modified to suit the particular experiment and the solvent is evaporated on a water-bath or *in vacuo*, leaving the crude material in a ring in the bottom of the container. Then, after placing a light ring of grease along the upper edge of the

(2) K. C. D. Hickman and C. R. Sandford, *J. Phys. Chem.*, **34**, 637 (1930).

(3) Othmar Werner, *Mikrochemie*, **1**, 33 (1923).

(4) W. H. Carothers and J. W. Hill, *THIS JOURNAL*, **54**, 1557 (1932).

(5) W. H. Strain and W. M. Allen, *Ind. Eng. Chem., Anal. Ed.*, **7**, 443 (1935).

(6) The Scientific Glass Apparatus Co., of Bloomfield, N. J., have satisfactorily made it for us.

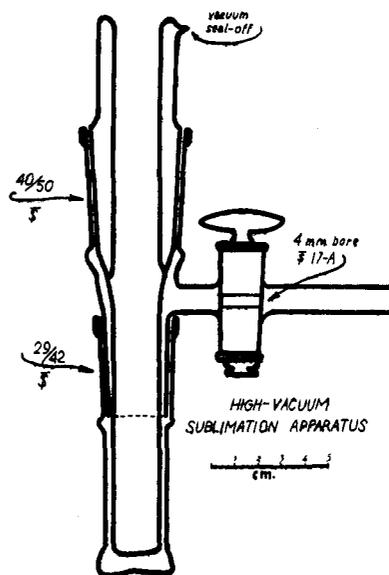


Fig. 1.

ground joint, the cup is slipped into place. The condenser is similarly greased and inserted. After evacuation of the apparatus the refrigerant is added. Water may be used, if desired, by inserting a stopper carrying one long and one short tube, and passing the water through these tubes. We have found that any of the low-temperature refrigerants, including liquid nitrogen, has been satisfactory. The lower cup is warmed by insertion into a bath at the desired temperature, and sublimation proceeds. When the process is completed, the sublimate can be washed from the condenser with a few drops of solvent, and worked up in any way desired.

CHICAGO, ILLINOIS

RECEIVED MARCH 12, 1938

The Effect of β -Aminopyridine in Experimental Blacktongue

BY Y. SUBBAROW, W. J. DANN AND E. MEILMAN

β -Aminopyridine was isolated from liver extracts by the following method.

Eight liters of solution (Cohn fraction D)¹ representing one ton of fresh liver was diluted to 20 liters with water and treated with 3 kg. of charcoal. The charcoal adsorbate was filtered and was eluted with hot 60% ethyl alcohol. The elute was concentrated to four liters. The concentrate was made strongly alkaline with potassium hydroxide and extracted three times with four liters of normal butyl alcohol each time.

(1) E. J. Cohn, G. R. Minot, G. A. Allen and W. T. Salter, *J. Biol. Chem.*, **77**, 331 (1928).

The butyl alcohol was removed from the extract by vacuum distillation, and the residue was taken up with one liter of water. There was some insoluble residue which was discarded.

The clear solution was made strongly alkaline with potassium hydroxide and was extracted eight times with one liter of ether each time. The ether extract was dried with anhydrous potassium carbonate and concentrated to a sirup. The sirup was extracted with 500 cc. of hot benzene. The benzene extract was concentrated to a small volume (30-40 cc.) and dry petroleum ether was added till a precipitate began to appear. After standing at 0° for forty-eight hours, a crystalline precipitate was obtained.

The crystals were filtered, and recrystallized from hot benzene and dried over sulfuric acid; yield 150 mg. The analysis agreed with the formula $C_8H_8N_2$.

The substance was hygroscopic and melted at 65°. Synthetic β -aminopyridine melted at 64°. There was no depression of mixed melting point. It gave an intense diazo reaction and was partially precipitated with cuprous oxide. It reduced phenol reagent (Folin). A gold salt formed readily by the addition of gold chloride in the presence of dilute hydrochloric acid, of composition $C_8H_8N_2 \cdot HAuCl_4$, m. p. 218°. The substance gave a flavianate. The crystals were rectangular plates. They began to char at 212° and melted with decomposition and evolution of gas at 241°. The flavianate of synthetic β -aminopyridine behaved in the same way. The picrate melted at 188-190°. There was no depression of mixed melting point with β -aminopyridine picrate. With concentrated hydrochloric acid, the substance gave transparent plates: β -aminopyridine dihydrochloride, $C_8H_8N_2 \cdot 2HCl$, m. p. 175°.

Six adult dogs weighing, respectively, 9.15, 12.3, 7.75, 15.1, 11.2 and 11.0 kg. were housed in individual cages and fed on Goldberger's blacktongue-producing diet No. 123² modified by substituting a more complete salt mixture for the sodium chloride and calcium carbonate used in this diet. The dogs were allowed to develop blacktongue, as judged by inflammation and necrosis of the mucous membranes of the mouth and throat, excessive salivation, listlessness, loss of appetite and loss of weight. After the onset of acute blacktongue each was given daily doses of a solution of β -aminopyridine dihydrochloride in distilled water for five days; administration was by subcutaneous injection. The daily dose for each of the six dogs was 20, 20, 20, 15 and 15 mg., respectively. The dihydrochloride contains 56.5% of the free base.

All the dogs quickly made complete recoveries, as shown by return of the mucous membranes to normal, cessation of the abnormal salivation, and restoration of appetite, body weight and normal activity.

The rapidity and completeness of cure were at least equal to the rapidity and completeness of cure of other dogs to which we have given 20 mg. of nicotinic acid daily for five days, showing that β -aminopyridine is, weight for weight, at least as active in curing blacktongue as nicotinic acid. Further experiments are in progress to determine whether smaller doses of β -aminopyridine will be completely effective in curing an attack of acute blacktongue, as there are indications from a preliminary trial that they may be.

(2) J. Goldberger, G. A. Wheeler, R. D. Lillie and L. M. Rogers, *U. S. Pub. Health Rep.*, **41**, 297 (1926).

The authors wish to thank Dr. Guy W. Clark and Dr. Merton C. Lockhart of Lederle Laboratory for help in large scale preparation, and the Milton and Proctor funds of Harvard University and the Rockefeller Foundation

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RECEIVED MARCH 29, 1938

COMMUNICATIONS TO THE EDITOR

COMPARISON OF X-RAY PHOTOGRAPHS TAKEN WITH X AND Y BUILT-UP FILMS

Sir:

Some time ago Mr. Clifford Holley and I reported [*Phys. Rev.*, **52**, 525 (1937)] that X films of calcium stearate have approximately the same grating-space as Y films of barium-copper stearate. In Figs. 1 and 2 are shown some of the photographs taken with films built by Dr. Katharine Blodgett on the basis of which this statement was made. They show $L\alpha$ and $L\beta$ lines of tungsten in the first three observable orders on both sides of the direct beam. Figure 1 was taken with an 1100 layer Y film of barium-copper stearate, Fig. 2 with a 300 layer X film of calcium stearate. In both cases the direction of dipping was parallel to the axis of rotation of the film. It can be seen that the photograph taken with the X film is essentially the same as the one taken with the Y film with respect to both the grating-space and the relative intensities of the various orders. The grating-spaces of these films, corrected for refraction and based upon the ruled grating wave length scale, are: Y film of barium-copper stearate, 50.47 Å.; X film of calcium stearate, 50.12 Å.

Porter and Wyman have shown [THIS JOURNAL, **59**, 2746 (1937)] that the contact potentials of X films increase with the number of layers, whereas those for Y films remain constant. The X-ray photographs, however, seem to be independent of the contact potential. The potentials of an X and a Y film of calcium stearate were measured, after which photographs of the kind shown here were taken. The photographs were much the same as those shown, despite the difference in the contact potentials of the films before exposure. The apparent grating-spaces of the two films in the first order differed by about 1%, the X film

having the greater spacing. The two films were built from 10^{-4} molar solutions of calcium chlo-

Fig. 1.—Three orders of tungsten L-spectra from Y film.



Fig. 2.—Three orders of tungsten L-spectra from X film.

ride. Castor oil was used as piston oil for the X film, and oleic acid for the Y film.

RYERSON PHYSICAL LABORATORY SEYMOUR BERNSTEIN
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RECEIVED APRIL 22, 1938

PRESSURE-AREA RELATIONS OF MONOLAYERS AT THE SOLID-LIQUID INTERFACE

Sir:

While the pressure $F = \gamma - \gamma'$ of a monolayer on water may be determined by a film balance, no method has been known for obtaining F at a solid-liquid interface. In order to determine this value the contact angle θ_1 between the plane surface of the solid and the pure liquid is found by an improved form of the tilting plate method, so arranged that the surface of the liquid can be kept clean by sweeping. Then the pure liquid of surface tension γ_1 , *e. g.*, water, is replaced by a solution of the desired solute, of tension γ_1' and the new contact angle θ_2 is determined. Then

$$\gamma_s = \gamma_{sl} - \gamma_1 \cos \theta_1 \quad (1)$$

$$\gamma_s' = \gamma_{sl}' - \gamma_1' \cos \theta_2 \quad (2)$$

where s indicates a solid.