TABLE V--BASICITY AND EXTRACTION POWER OF WEAK NITRIC ACID

Material Extracted	Phosphorus Extracted Per cent
0.1 g. Rock Phosphate.....	8.555
0.1 g. Rock Phosphate + 0.1 g. Calcium Carbonate.....	8.413
0.1 g. Rock Phosphate + 0.2 g. Calcium Carbonate.....	8.362

The percentages of phosphorus extracted would have been higher had a larger volume of the nitric acid been employed for the extraction. Table VI shows that with various larger volumes of solvent, practically identical percentages of the phosphorus were dissolved, in all cases approaching the total phosphorus content of the rock phosphate, 12.37 per cent. The strength of the acid used was 0.01 per cent. The time for extraction was 30 min. as before and the amount of rock phosphate treated, 0.2 g.

Volume of Acid Used Cc.	Phosphorus Extracted Per cent
500	11.094
1000	12.150
1500	12.124
2000	12.131

SUMMARY

Summing up our results as to the solubility of the phosphorus of rock phosphate in very dilute citric and nitric acids with a view to obtaining an indication as to the available phosphorus in this particular

material, we find that both possess too high a solvent power.

An additional objection to the use of citric acid is that basicity has a decidedly depressing influence on the solvent power and this cannot be altogether overcome by the addition of an extra amount of citric acid equivalent to the basicity present.

While neutral ammonium citrate cannot serve as a means for measuring the absolute amount of available phosphorus, it probably gives as reliable an indication of the available phosphorus of rock phosphate and eventual changes in availability due to sulfonation and other processes as any solvent can be expected to furnish. It is necessary, however, that two determinations be made, one at the beginning and one at the end of the experiment, and absolutely uniform conditions maintained in both instances.

This latter point must be observed because many factors, including the amounts of the material taken and the volume of solvent, will appreciably affect the availability of phosphorus as measured by neutral ammonium citrate.

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THE RECOVERY OF WASTE PARAFFINED PAPER BY EXTRACTION WITH VOLATILE SOLVENTS

By OTTO KRESS AND L. F. HAWLEY

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Some time ago the Forest Products Laboratory was requested to determine whether there was a suitable method for recovering the paraffin and paper stock from waste paraffined paper. This waste paraffined paper has only a very limited value and in the majority of cases it is burned under the boilers. Other forms of paper waste such as oiled paper, asphalt-impregnated papers used for wrapping and case lining, board made damp-tight by means of asphalt or tar for the manufacture of cartons and containers of washing powder, coffee, etc., have very little application either, and are usually burned. Occasionally a small amount of the waste containing asphalt, not to exceed 2 per cent, is worked up in the beater with fresh paper stock, but the difficulties experienced with the pitch on the wire and presses of the paper machine, and the consequent loss of production due to breaks and the necessity of frequent clean-ups, makes the desirability of the practice very questionable.

No accurate statistics are available on the production of paraffined paper, but from a number of sources it is estimated that the production is close to 2,400,000 lbs. per week. Assuming a 4 per cent waste, there would be available 48 tons per week. In the above estimate no account has been taken of the waste incident to the manufacture of paraffined drinking cups, food containers, wrappers for special foods such as breakfast foods, etc.

From a review of the literature and from the experience of the manufacturer who brought the problem to us, it was believed that an attempt to remove the paraffin by melting it with hot water would not be successful. Several runs were made in which the paraffined paper was shredded and then opened up in a beater, the contents of which could be heated by the direct introduction of live steam. By this treatment it was possible to remove part of the molten wax by skimming and part by means of a washer, but the disintegrated pulp apparently held on tenaciously to a portion of the wax.

The resulting pulp was run off in the form of paper on our experimental paper machine, and an analysis for the percentage of wax in the finished paper is given for Run 7 in Table I. While the pulp prepared by this hot water treatment could be run over our experimental machine into finished paper, we believe, in view of the high wax content of the pulp, that it could not run for any length of time on a commercial machine. It was found from our experiment that the treatment of paraffined stock with hot water, heated by means of live steam, caused a decrease in the paraffin content to 0.90 per cent. In this experiment, however, the steaming operation was continued for 2 days, the condensed water carrying with it the extracted paraffin, overflowing constantly from the tub in which the stock was heated. A treatment of this nature, requiring so much time and steam expenditure, would not be commercially feasible.

Our coöperator supplied us with several hundred pounds of waste paraffin paper, some of which had

been printed previous to paraffining. Seven runs were made in our experimental extraction apparatus, using gasoline as the solvent, with the object of recovering the paraffin and waste paper stock and determining the general conditions of extraction.

The apparatus used for the extraction experiments consisted of a vertical cylindrical extractor 3 ft. high and 1½ ft. in diameter, provided with a closed steam coil and a steam jet. This extractor was connected with a condenser in such a manner that the vapors of solvent, when boiled off, could be led to the condenser, condensed, and returned again to the extractor, all in a closed system. In the experiments a weighed amount of the paraffined paper was placed in the extractor and a measured amount of the solvent (a middle fraction of gasoline, b. p. 90–140° C.) was added; then the cover was put on and by means of the closed steam coils the solvent was boiled slowly through the condenser, returning continuously to the extractor. The steam was then turned off and the solution drained from the bottom of the extractor. An amount of fresh solvent equal to that drained off was applied and the process repeated. The solvent remaining after the second extraction and draining was recovered by blowing live steam through the extractor to a condenser and collecting the mixed condensate of water and solvent.

Different proportions of solvent and paper and charges of different weights of paper per extractor were used in order to determine the best conditions for extraction. The conditions used and the results obtained are shown in Table I. In Runs 1 to 4 more solvent was used than was found to be necessary, that is, the small amount of paraffin left on the paper was apparently a minimum uninfluenced by the amount of solvent used. Runs 5 and 6 were made to determine how little solvent could be used and satisfactory extractions still be obtained. The maximum amount of paper which could be charged into the tractor was also determined in these runs. In Run 5 the paper scraps, as received in irregular, fairly large sized pieces, were packed directly into the extractor, with occasional tearing of the larger sheets or breaking up of the matted lumps. The material used in Run 6 was run through a Williams shredder; and as may be seen from the table, a larger amount of the shredded paper can be charged in a given space than of the larger sized scrap.

As would be expected, a larger amount of paraffin was left on the paper after extraction in Runs 5 and 6, where much smaller proportions of solvent were used than in the previous runs. It must be remembered that the method used in these experimental extractions would not be used in a commercial process, since in a commercial process where a large number of charges are to be extracted in just the same way, it is preferable to use the countercurrent system of extraction, in which one lot of solvent may be used in regular order on several different charges of paper. By such a method it would be possible to obtain a more complete extraction with the use of even less solvent.¹

¹ For complete consideration of this and other points of the extraction process, the reader is referred to the article, "Discontinuous Extraction Processes," by L. F. Hawley, *THIS JOURNAL*, 9 (1917), 886.

Under the conditions used in Run 6, the solvent ratio is 5/3, since, of the 8 gal. added to the paper, 5 gal. are drained off and 3 gal. are retained after draining. By using solvent at the rate of 8 gal. per 20 lbs. of paper in a series of 4 extractions, according to the countercurrent principle, it should be readily possible to obtain a 97.0 to 97.5 per cent extraction (theoretical, 98.02). If it were found that extraction in this way left too much paraffin in the extracted paper, an increase in the amount of solvent used would give more complete extraction. For instance, 12 gal. of solvent to 20 lbs. of paper would give a solvent ratio of 4 which in 4 treatments in series would give a theoretical extraction of 99.61 per cent.

These experiments have determined the conditions under which various degrees of extraction can be obtained, but which of these conditions would be most desirable in commercial practice can be determined only from a study of the costs of different parts of the process. For instance, let us compare the conditions outlined above, of 20 lbs. of shredded paper per 3 cu. ft. of extractor space and 8 gal. of solvent per 20 lbs. of paper, with another set of conditions such as those of Run 5, viz., 15 lbs. unshredded paper per 3 cu. ft. of space and 10 gal. of solvent per 15 lbs. of paper. In the latter case the unshredded and loosely packed paper retains less solvent; the solvent ratio is therefore greater and the extraction percentage which can be obtained in the same number of treatments is higher. The disadvantages of these conditions are a decreased capacity of apparatus and an increased cost of handling the larger amount of solvent (including probably larger solvent loss). These advantages and disadvantages must be compared in terms of costs before the most desirable conditions can be decided upon.

TABLE I
— SOLVENT — PERCENTAGE PARAFFIN
In Extracted Paper

RUN No.	Paper Used, Lbs.	Gal. Used at One Time	Gal. Retained after Draining	Solvent Ratio $a = \frac{\text{Col. 3}}{\text{Col. 4}}$	In Original Paper	On Basis Extracted Paper	On Basis Original Paper	Theoretical $\frac{1}{(\text{Col. 5} + 1)^2} \times \text{Col. 6}$
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)
1.....	7	20	0.65	30	25.5	0.21	0.15	0.03
2.....	11	16	1.1	15	26.8	0.24	0.16	0.11
3.....	11	16	1.0	15	23.5	..	..	0.09
4.....	11	16	1.2	4	19.5	0.46	0.35	0.08
5.....	15	10	2.1	4	19.5	2.03	1.67	0.86
6.....	20	8	3.0	5/3	19.1	3.40	2.67	2.67
7.....	..	..	..	..	..	7.90	..	..

Our coöperators supplied us with some samples of their mill run of paper before impregnation in order that its strength might be compared with that of the paper made from the extracted pulp. Sample 1 was made at the mill on the following furnish:

50 per cent bleached sulfite pulp
25 per cent bleached soda pulp
25 per cent unbleached sulfite pulp

Sample 2:

20 per cent bleached soda pulp
80 per cent bleached sulfite pulp

All the extracted pulps were made into paper on our experimental paper machine and it may be of interest to compare (Table II) a paper made on Run 7

with the original unimpregnated paper. The paper made from the extracted pulp from Run 7 had the poorest pulp in so far as thoroughness of paraffin extraction was concerned. The strength data clearly indicates that the extraction process had no decided influence on the strength of the pulp.

Some experiments were also made at an earlier date at the request of a manufacturer of damp-tight cartons and packages to determine whether the waste paper scraps from his plant might be treated to recover the paper stock. The damp-tight carton stock consisted of two boards cemented together with asphalt or tar to render the board impervious to moisture. The scrap as received contained 6.6 per cent moisture and 8.6 per cent of ether-soluble material. After treatment in the laboratory extractor the paper residue was made into board on our experimental machine and showed no spots of unextracted material. The extracted paper scrap showed an ether-soluble extract of 0.76 per cent.

TABLE II—STRENGTH TESTS ON COMMERCIAL SAMPLES 1 AND 2, AND CALENDERED SHEET FROM RUN 7. TESTS MADE AT 65 PER CENT HUMIDITY AND 76° F.

	Weight per Ream 24 × 36/300 Lbs.	Mullen Points	Strength Ratio per lb. Points	Factor per 0.001 Thickness Points	Breaking Length Average Meters	Folds Aver- age No.	Stretch Average Per cent
Sample 1.....	25.5	7.4	0.290	3.7	3010	4	1.27
Sample 2.....	26.5	5.3	0.20	2.62	1910	3	1.27
Run 7.....	32.0	9.2	0.287	3.03	2330	4	1.43

Some of the paraffined waste paper had been printed with advertisements in a blue ink previous to impregnation. It was found that the solvent extraction of the wax did not affect the blue ink, nor did bleaching with ordinary bleaching powder after the extraction and pulping of the paper. This ink could be removed by means of an alkaline treatment of hydrosulfite and subsequent washing, but reoxidation to the original blue color occurred in the air unless the reduced and consequently soluble dyestuff was removed by washing. No examination was made to determine the particular pigment employed, but from the general reaction there is no question that it was of the nature of a vat dye similar to the indanthrenes. For the printing of bread wrappers, where no great permanency of the printed matter is required, cheaper printing inks which could be readily bleached with chlorine might be used with a consequent saving in cost and ease of bleaching of the resulting pulp.

From the above, it can readily be seen that the extraction of waste paraffined paper by means of gasoline and the consequent saving of the pulp and wax can be accomplished without undue difficulties. The resulting paper stock carries no more material soluble on extraction with petroleum ether than will be found in the form of pitch and resin in good grades of commercial sulfite pulp. Further, the paper stock is not injured, and, with possibly a slight bleaching treatment, may be used again for the manufacture of waxing paper.

The main difficulty attending such a recovery project lies in the fact that the amount of waste available in the United States is just about sufficient for the operation of a medium-sized extraction plant. With the constant increase, however, in the use of paraffined

paper for the manufacture of bread wrappers, food containers, etc., the extraction of the paper waste and the recovery of the paraffin, if handled at a centralized plant, may well become a profitable conservation.

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AN IMPROVED AUTOMATIC BURETTE

By GEORGE J. HOUGH

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The burette herewith illustrated was devised by the writer several years ago, and has been found very handy and convenient. Its principal merits are, first, that it requires no bracket or support to hold it on the solution reservoir, and second, that it can be quickly cleaned and used for some other solution; this I have found to be a great advantage as only one burette is required for a large number of standard solutions, especially solutions that are not in constant use, thus obviating a number of permanently attached burettes.

As is seen in the illustration, the base of the burette consists of a glass tube, *a*, for elevating the solution, surrounded by an outer jacket consisting of a much wider tube; and when inserted in a rubber stopper to fit the solution reservoir, this form of base gives the apparatus sufficient stability so that it requires no bracket to hold it upright. The tube *c* is for the attachment of a rubber pressure bulb, and tube *d* is closed with the finger when pumping air into the reservoir. The hole in the outer jacket of the base is to equalize the pressure in the reservoir. The other details of the apparatus require no explanation.

The dimensions of the apparatus are as follows: the burette *e*, including lower stopcock, is 24 in. long; the outlet tube is 4 in. long; jacket *a* is 10.5 in. long, with a diameter of 0.5 in.; tube *c* is 2.25 in. long, and tube *d* is 1 in.; the distance from the top of jacket to the hole *h* is 1.75 in. This hole must be above the level of the liquid in the reservoir.

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