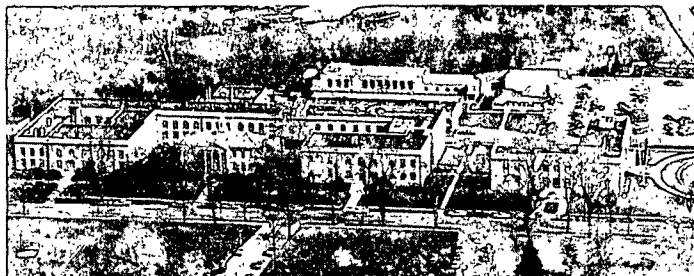


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DEVELOPMENT OF A MANUFACTURING PROCEDURE FOR LOW-LITHIUM,
LOW-URANIUM CONTENT FILTER PAPER

Project 3101

Report Four

A Status Report

to

DEPARTMENT OF THE AIR FORCE
1155th TECHNICAL OPERATIONS SQUADRON (HQ. COMD.)
McCLELLAN AFB, CALIFORNIA

March 6, 1973

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

DEVELOPMENT OF A MANUFACTURING PROCEDURE FOR LOW-LITHIUM,
LOW-URANIUM CONTENT FILTER PAPER

SUMMARY

The analysis of Samples 3101-026 to 3101-096 was completed by McClellan AFB, MCL-C. The results support the conclusion that processes are available for reaching the goal of < 0.3 ng. U/g. of pulp using the Hercules cotton linter, PS-57. Comparable levels of uranium are more difficult to reach with IPC-1478 paper (without Kronisol) than with the Hercules pulp, but one lot of such paper processed at McClellan AFB was well-within the goal.

Blotter stock used in the making of handsheets at the IPC, and known to contain much more lithium and uranium than purified linter pulps, was treated with hydrofluoric acid. The lithium and uranium contents were lowered to 1.4 ng. Li and 0.6 ng. U/g.

The cotton linters pulp (Hercules PS-57) was treated with lithium-6 ($> 99\%$ enrichment) and then acid-washed. The results indicated that the lithium which was inaccessible to the acid was not exchangeable with the lithium-6.

The cotton linter pulp was leached with hydrofluoric acid in concentration from 2 to 0.002%, but neither the lithium nor uranium contents of the acid-washed pulps appeared to be related to the concentration of the acid.

Encasing the cotton linter pulp, flooded with dilute hydrofluoric acid, in a dialysis casing and then immersing the casing in a slurry of an Amberlite mixed-bed resin achieved approximately the same result as the procedure commonly used for acid-washing such pulp.

IPC-1478 filter paper was washed with 0.2 and 0.02% hydrofluoric acid.
The 0.2% acid was more effective than the 0.02%, but only the uranium was lowered
to the specified level with the stronger acid.

INTRODUCTION

The analytical results for the samples described in Report Three (1) contained only a scattering of results for uranium. The data are now complete and all the data available are listed in Tables I and III. The data in Table II were obtained for an additional set of samples.

TABLE I
LITHIUM CONTENT OF IPC SAMPLES
(3rd Shipment)^a

IPC Sample No.	Description ^b	Lithium		Uranium		
		ng./g.	ng./ml.	8/5	ng./g.	ng./ml.
3101-026	Deionized water from tap in Room 209	A (teflon beaker)	0.79	129		0.00082
		B (Pt dish)	1.06	135		0.00056
-027	Double-deionized water (effluent from a cartridge of mixed bed resin fed from deionized water tap in Rm. 209)	-- ^c	0.021	126		0.00068
-028	2% nitric acid reagent		0.014	119		0.0010
-029	Filtrate (1 liter) from 2% nitric acid treatment of pulp (030-034)	A (teflon beaker)	0.14	115		0.087
		B (Pt dish)	0.14	115		0.069
-035	Water washings (1 liter) from pulp after 2% nitric acid leaching		0.013	125		0.0025
-030	Circle 1 (top) Circle 2 Stack of pulp circles (73.7 g.) leached with 2% nitric acid; washed with water; dried		5.33	138	6.50	
			1.58	133	0.22	
-032	Circle 6 Circle 7 Circle 8		0.78	123	0.97	
			1.21	138	0.113	
			1.15	130	0.209	
-034	Circle 11 Circle 12 (bottom)		0.90	135	0.269	
			1.62	125	0.123	
-036	2% hydrochloric acid reagent		0.017	134		0.0011
-037	Filtrate from 2% hydrochloric acid treatment of pulp		0.144	87		0.072
-043	Water washings from pulp (038-042) after 2% hydrochloric acid leaching	A (teflon beaker)	0.015	127		0.0018
		B (Pt dish)	0.015	127		0.0015
-038	Circle 1 (top) Circle 2 Stack of pulp circles (74.0 g.) leached with 2% hydrochloric acid; washed with water; dried		2.67	130	2.10	
			1.00	127	0.157	
-040	Circle 6 Circle 7 Circle 8		1.19	126	0.133	
			1.17	127	0.138	
			1.01	128	0.128	
-042	Circle 11 Circle 12 Circle 13 (bottom)		1.56	125	0.102	
			1.17	124	0.097	
			1.19	127	0.129	
-044	2% hydrofluoric acid reagent		0.018	136		0.0036
-045	Filtrate from 2% hydrofluoric acid treatment of pulp (046-050)		0.234	116		0.139

^a See end of table for footnote.

TABLE I (Continued)
 LITHIUM CONTENT OF IPC SAMPLES
 (3rd Shipment)^a

IPC Sample No.	Description ^b	Lithium		Uranium		
		ng./g.	ng./ml.	8/5	ng./g.	ng./ml.
-051	Water washings from pulp after 2% hydrofluoric acid leaching		0.018	123		0.0084
-046 Circle 1 (top)	Stack of pulp circles (111.1 g.) leached with 2% hydrofluoric acid; washed with water; dried	2.56		136	0.998	
		0.89		127	0.214	
-048 Circle 9		0.69		128	0.238	
Circle 10		0.71		85	0.168	
Circle 11		0.79		123	0.162	
-050 Circle 18		0.59		121	0.157	
Circle 19		0.66		120	0.161	
Circle 20 (bottom)		0.79		122	0.206	
-052	Circles of pulp leached with 2% hydrofluoric acid followed by 0.05% hydrofluoric acid; not dried before shipment; dried at MCL	0.90	A	110	0.260	
		1.01	B	91	0.279	
		1.12	C	115	3.575	
-053	Water filtrate (2 l.) from the dispersion of 260 g. of pulp in 4 l. of water		0.20	59		0.0077
-054	Filtrate (2 l.) from 2% hydrofluoric acid leaching of 260 g. pulp (056-058)		0.117	115		0.114
-055	Water effluent from purified pulp (056-058)		0.013	(36)		0.00053
-056 Top zone	Hercules PS-57 pulp, 260 g., leached with 2% hydrofluoric acid; washed with water; dried	A	1.23	120	0.293	
		B	1.19	95	0.301	
-057 Middle zone		A	1.04	124	0.348	
		B	0.79	118	0.254	
-058 Bottom zone		A	0.56	113	0.153	
		B	0.48	113	0.126	

^aAll analytical data were obtained at the McClellan AFB, MCL-C, and were transmitted in letters dated December 21, 1972 and February 5, 1973, from Captain James R. Wright to E. E. Dickey.

^bIn this report "pulp" refers to Hercules PS-57 cotton linters.

^cAll unspecified analyses were ashed in platinum dishes.

TABLE II
LITHIUM CONTENT OF SAMPLES OF
THE INSTITUTE OF PAPER CHEMISTRY
(Fourth Shipment)^a

IPC Sample No.	Description	Lithium				Uranium		
		Main ^c	Spiked ^d	ng./g.	ng./ml.	8/5	ng./g.	ng./ml.
3101-059	2% HF filtrate (1 liter) from blotter stock ^b				1.103	138		1.002
-060	1 (top), 2			2.00 (top)		137	0.787	
-062	Circle 6	A		11.5		137	0.588	
	Circle 7	B		1.35		137	0.625	
	Circle 8	C		-- ^b		138	0.581	
-063	Circle 9			1.36		138	0.597	
	Circle 10							
	Circle 11							
-066	Circle 19			-- ^b		139	0.638	
	Circle 20							
	Circle 21 (bottom)							
-067	Blotter stock, control ^b	A		-- ^b		140	17.6	
		B		-- ^b		138	17.0	
-069	Purified pulp exposed to 4 ng./g. of lithium-6	A	2.94	9.13	3.86	120	0.383	
		B	3.46	9.80	4.12	121	0.411	
-073	Water filtrate (4 l.) from pulp (258 g.)		0.0764	3.37		0.086	115	0.0040
-074	2% hydrofluoric acid reagent		0.0745	0.906		0.341	133	0.0085
-075	2% hydrofluoric acid filtrate (3.6 l.)		0.0886	0.463		0.765	118	0.0742
-076	Reagent for lithium-6		18.72	114.7		0.161	92	0.00035
-077	Filtrate (3.8 l.) from lithium-6 treatment		2.653	56.4		0.0060	114	0.00062
-079	Purified pulp	A	0.0867	6.15	1.11	116	0.394	
		B	0.0784	6.39	1.04	122	0.380	
-080	Lithium-6 pulp (3101-069), leached with water	A	0.868	8.73	1.75	129	0.676	
		B	1.430	5.47	3.24	131	1.102	
-081	Lithium-6 pulp (3101-069), leached with 2% HF; water	A	0.098	7.69	1.03	123	0.0679	
		B	0.097	6.19	1.12	131	0.0806	
-082	2% HF filtrate from 20 g. of treated pulp (3101-081, 1 liter)		0.218	(0.662)		0.712	129	0.0179

TABLE II (Continued)
 LITHIUM CONTENT OF SAMPLES OF
 THE INSTITUTE OF PAPER CHEMISTRY
 (Fourth Shipment)^a

IPC Sample No.	Description		Lithium				Uranium	
			6/7		ng./g.	ng./ml.	8/5	ng./g.
Main ^c	Spiked ^d							
-083	Pulp leached with 2% HF	A			0.682	129	0.856	
		B			0.635	118	0.343	
-084	Pulp leached with 0.2% HF	A			0.639	116	0.316	
		B			0.633	116	0.306	
-085	Pulp leached with 0.02% HF	A			0.506	116	0.427	
		B			0.441	116	0.337	
-086	Pulp leached with 0.002% HF	A			0.593	118	0.258	
		B			0.458	117	0.207	
-087	Pulp leached with 2-0.002% HF	A			0.616	116	0.373	
		B			0.714	115	0.379	
-089	Pulp + 0.4% hydrofluoric acid in dialysis casing; dialyzed against Amberlite MB-3	A			0.637	123	0.355	
		B			0.684	127	0.418	
-092	IPC-1478, without Kronisol, leached with 0.2% HF	A			2.78	135	0.270	
		B			2.95	134	0.272	
-096	IPC-1478, without Kronisol, leached with 0.02% HF	A			4.10	137	0.397	
		B			4.15	136	0.376	

^aAll analytical data were obtained at the McClellan AFB, MCL-C, and were transmitted by letters dated February 5 and 20, 1973, from Captain James R. Wright to E. E. Dickey.

^bLithium analyses were not successful for these samples.

^cAtom ratio of the isotopes, Li-6/Li-7, in the sample as received.

^dAtom ratio of the isotopes, Li-6/Li-7, in the sample after 18×10^{12} atoms of Li-6 have been added to the analytical sample.

TABLE III

LITHIUM CONTENT OF IPC-1478 FILTER PAPER^a

Experiments and analyses performed at the McClellan AFB, MCL-C, on paper (untreated with Kronisol) after chemical purification

MCL-C No.	Description		Lithium	Uranium	
			ng./g.	8/5	ng./g.
Control	June, 1970		7.53	--	--
012-5538 control	November, 1972	A	8.62	133	0.830
		B	8.95	134	0.877
012-5539	Scrim removed; stack of ten circles leached with hydrochloric acid at pH 3, water-washed, leached with ammonium carbonate at pH 9, water-washed, dried	A	4.71	126	0.359
		B	(7.96)	(99)	(0.608)
		C	4.32	107	0.329
		D	5.00	109	0.349
		E	4.32	126	0.330
		F	5.36	(55)	0.408
		G	4.93	117	0.361
		H	5.44	106	0.343
		I	4.82	125	0.370
		J	4.74	124	0.383
012-5540	Same experimental conditions as in 012-5539 above except that the scrim was removed after processing the circles and just prior to dry ash analysis	A	4.93	--	--
		B	4.80	130	0.424
		C	4.60	133	0.453
		D	4.87	133	0.516
		E	5.87	134	0.653
		F	5.12	126	0.463
		G	5.10	132	0.514
		H	5.50	133	0.520
		I	4.02	131	0.475
		J	4.18	133	0.455
	Process water, double-distilled		0.015 ng./ml.		
012-5541	Control	A	5.26	123	0.397
		B	5.96	(71)	0.795
	Stack of sixteen circles leached with ammonium carbonate pH 9, water-washed, leached with hydrochloric acid at pH 3, water-washed, dried	C	3.67	117	0.288
		D	2.63	127	0.266
		E	2.90	124	0.164
		F	1.99	127	0.167
		G	2.07	127	0.236
		H	2.25	121	0.189
		I	2.13	107	0.166
		J	5.98	128	0.233
		K	1.84	127	0.164
		L	2.20	87	0.194
		M	2.11	122	0.138
		N	1.98	128	0.188
		P	1.80	129	0.251
		R	2.33	129	0.230
S	2.90	133	0.258		
T	2.56	129	0.243		

^aThe data were obtained at the McClellan AFB, MCL-C, and were transmitted in a letter dated February 5, 1973 from Captain James R. Wright to E. E. Dickey.

DISCUSSION

GENERAL

By inspection of the data in Table I, it seems clear that portions of both lithium and uranium can be removed readily with dilute acids from cotton linter pulp, Hercules PS-57¹. However, lithium levels of < 0.4 ng. Li/g. of pulp were not attained; the lowest value was 0.48 ng./g. (3101-058). On the other hand, uranium was consistently < 0.3 ng. U/g. of pulp, and with many samples < 0.2 ng./g. In most cases the 8/5 ratios were 120-130 and indicated that the residue was a mixture of the uranium ($8/5 = 116$) contained in the original pulp and the more common natural uranium ($8/5 = 136$) from the reagents and the process water. It may be noteworthy that the uranium in Sample 3101-058 appeared to be only a residue from the original pulp ($8/5 = 113$). The upper zones of pulp (3101-056 and -057) in this experiment may have scavenged the small amount of uranium present in the process water. If it is assumed that the behavior of lithium is similar to that of uranium, the residual lithium may represent the fraction of the original lithium not accessible to the hydrofluoric acid. Therefore, techniques other than leaching with hydrofluoric acid alone may be required in approaching the goal of < 0.4 ng. Li/g. of pulp.

With these data and assumptions in mind, experiments were performed which involved the labeling of pulp with lithium-6 followed by leaching with hydrofluoric acid. As shown in Table II a sample of acid-washed pulp (3101-079) was exposed to 4 ng. of lithium-6 per gram of pulp (3101-069); the absorption of lithium-6 was essentially quantitative². Subsequent leaching of the labeled

¹See Report Three for additional discussion.

²See also the lithium-6 reagent (3101-076) and the filtrate (3101-077) from the lithium-6 treatment of the acid-washed pulp.

pulp with hydrofluoric acid (3101-081) reduced the lithium content to 1 ng. Li/g. with a 6/7 ratio only slightly higher than that of natural lithium, indicating that the lithium in the acid-washed pulp was largely inaccessible to the acid or to other lithium ions. As might be expected, water alone removed less than half the sorbed lithium (3101-080).

In addition to the use of a pulp labeled with lithium-6, a series of experiments (3101-083 to -087, Table II) was designed to test the possible relationship between metal-removal and concentration of acid.

The Hercules linter pulp (PS-57) was leached with four concentrations of hydrofluoric acid (2 to 0.002%), but neither the lithium nor uranium contents of the acid-washed pulps appeared to be related to the concentration of the acid. However, IPC-1478 paper leached with two concentrations of hydrofluoric acid (3101-092 and -093) showed some improvement of lithium and uranium removal by the higher concentration of acid. Additional work may be necessary to determine the most useful concentration of acid in these applications.

Because acid-washed pulps readily sorb lithium and uranium from aqueous solutions as indicated by the sorption of lithium-6 (see above), the following experiment was performed. Hercules PS-57 pulp was exposed to a suspension of a mixed-bed ion-exchange resin (Amberlite MB-3, Mallinckrodt AR). By this method we hoped to attain lower levels of lithium than had been achieved by more conventional procedures of acid-washing. The pulp was flooded with 0.4% hydrofluoric acid and encased in a cellulose dialysis casing to exclude resin particles from the pulp. After standing overnight, the pulp (3101-089) was neutral to Congo Red paper. However, the lithium and uranium contents were approximately

the same as those (3101-083 to -087) treated in a more conventional manner, and, for the present, this method appears to have no advantage over other methods.

IPC-1478 FILTER PAPER PROCESSED AT McCLELLAN AFB, MCL-C

Samples of IPC-1478 filter paper (untreated with Kronisol) were treated with hydrochloric acid and ammonium carbonate (Table III). When leached with acid followed by ammonium carbonate with intermediate and final water washing, Samples 012-5539 and 012-5540 showed that both lithium and uranium had been lowered somewhat. These results are consistent with those obtained previously for Sample 012-5537.(2). When the order of addition of chemical agents was reversed (Sample 012-5541), the process was apparently more effective in removing both metals with the uranium content substantially below the prescribed level of < 0.3 ng. U/g.

DEIONIZED WATER

As indicated in Report Three (1), the effluent from a back-up cartridge of mixed-bed resin was led into a special high-capacity mixed-bed resin and polished with an adsorption resin to remove colloidal silica and hydrous metal oxides¹. Because lithium is the most readily displaced metal in the displacement series, this cation may be expected to appear ahead of other cations in the effluent from an ion-exchange system as the resins approach saturation. However, it is hoped that the treatment as described may lower the lithium content of process water to < 0.010 ng./ml. At this level, process water could supply no more than 10 ng. of lithium per liter for possible sorption by chemically treated pulps.

¹Water purification resins: Research Cartridge No. 1506-30 and Puritan Cartridge No. 150-640, Cole Parmer Instrument Co., Chicago, Illinois.

This would expose 50 g. of pulp in 1 liter of wash water to < 0.2 ng. Li/g.
Therefore, if the treatment prior to the washing step reduces the lithium content to < 0.2 ng./g., the final product would average < 0.4 ng. Li/g., the specified maximum sought. Thus far, process water has exceeded the suggested maximum (3101-027, -055, Table I). In an effort to circumvent this problem, the percolation technique for treating and washing stacks of dry-lapped pulp and circles of IPC-1478 paper has been used with water-to-fiber ratios of 200 g. to 400 g. per liter instead of 20 g. to 50 g. per liter. The experiments are in progress and will be described in future reports.

BLOTTER STOCK LEACHED WITH HYDROFLUORIC ACID

Blotters used by the IPC in making handsheets were cut into circles and leached with hydrofluoric acid. These blotters were known to be considerably higher in both lithium and uranium than the linter pulps. The purified blotter circles (3101-060 to -066) and the acidic filtrate (3101-059) were analyzed. The results, Table II, indicated that 2% hydrofluoric is capable of producing blotters with lithium and uranium contents comparable with those of the purified Hercules pulp, PS-57. Blotters processed in this way may be useful in making IPC-1478 filter paper from a purified linter pulp such as Sample 3101-058 (Table I).

FUTURE WORK

1. Experiments are under way with the Hercules PS-57 pulp and with IPC-1478 paper to test the possibility of displacing lithium with sodium.
2. Based on the success achieved at the McClellan AFB, MCL-C with ammonium carbonate and hydrochloric acid, experiments are under way to adapt the method to hydrofluoric acid treatments.
3. Experiments are under way to test the practicability of leaching stacks of dry-lapped pulp in the form of disks (octagons) with chemical agents and with 200 to 400 g. of pulp/liter of aqueous solution instead of 20 g. to 50 g./liter as commonly used. Lithium-6 was added to some samples to test the accessibility and exchangeability of the lithium.

EXPERIMENTAL

Experimental procedures for Samples 3101-026 to -058, Table I, were described in Report Three (1) and are not repeated in this report. The reader may now ignore Table I, Report Three. Table I of Report Four (this report) summarizes all the analytical data for these samples, 3101-026 to -058. The samples listed in Table II were prepared by procedures reported herein. The detailed procedures for the samples listed in Table III may be procured from McClellan AFB, MCL-C.

BLOTTER STOCK LEACHED WITH HYDROFLUORIC ACID

A stack of 21 circles (59.8 g.) of blotter stock was placed on the funnel and 1.0 liter of 2% hydrofluoric acid was percolated through the stack. The percolation of the acid was interrupted three times to press the stack with a rubber dam in order to diminish any tendency toward bubble formation and channeling. Percolation was slow and a partial vacuum was used to maintain a satisfactory flow of liquid through the stack of blotter circles. The first portion of filtrate was yellow colored, and was collected (1 liter) for analysis (3101-059). The circles were washed, pressed, and dried in the usual way (Table IV).

LABELING PULP WITH LITHIUM-6

Purification of Pulp

An amount of 258 g. of pulp (Hercules, PS-57) was dispersed in water in two portions. The first portion was collected in the polyethylene funnel described previously (1) and the filtrate was used with some additional water for the dispersal of the second portion. The total amount of aqueous filtrate was 4 liters and was

designated 3101-073. The mat (column) of pulp was leached by percolation under gravity with 2 liters of 2% hydrofluoric acid followed by 3 liters of water. The wet pulp was divided into three portions by weight, airdry basis; (1) 46 g., (2) 28 g., designated 3101-079, and (3) 184 g. The 2% hydrofluoric acid reagent was designated 3101-074, and the combined acid filtrate and washings, 3.6 liters, was designated 3101-075.

TABLE IV
PURIFIED BLOTTER SAMPLES

Sample No.	Description
3101-059	2% hydrofluoric acid filtrate (1 liter) from 59.8 g. of blotter stock
-060	Circles 1 (top), 2
-061 ^a	Circles 3, 4, 5
-062	Circles 6, 7, 8
-063	Circles 9, 10, 11; these circles not marked nor dried
-064 ^a	Circles 12, 13, 14, 15
-065 ^a	Circles 16, 17, 18
-066	Circles 19, 20, 21 (bottom)
-067	Original blotter stock, control

^aNot submitted for analysis; retained by IPC.

Treatment with Lithium-6

The third portion of purified pulp, 184 g., was dispersed in 3 liters of water containing 736 mg. of lithium-6 in the form of lithium-6 carbonate, > 99% enrichment.¹ After 90 minutes with occasional stirring by hand, the pulp was recovered in eight arbitrary pads on the funnel without washing, each was pressed under a rubber dam, and dried overnight at 50° in an oven with gravity circulation of air. Pads No. 4 and 5 were combined (total 27 g.) and designated 3101-069, and served as the lithium-6 control pulp. A portion of the lithium-6 reagent was designated 3101-076, and the aqueous filtrate, 3.8 liters, from treating the pulp, 3101-077.

ATTEMPTED REMOVAL OF LITHIUM FROM LITHIUM-TREATED PULP

Leaching with Water

An amount of 10 g. of the ⁶Li-treated pulp (3101-069) was dispersed in 1.0 liter of water, leached for 60 min. with occasional stirring by hand, filtered, pressed, and dried as Sample 3101-080.

Leaching with Hydrofluoric Acid

An amount of 10 g. of the ⁶Li-treated pulp (3101-069) was leached with 250 ml. of 2% hydrofluoric acid for 30 min., the slurry was filtered, the pulp was washed with 300 ml. of water, pressed and dried as Sample 3101-081. The combined filtrate and washings from 20 g. of ⁶Li-pulp leached with acid was designated 3101-082.

¹Lithium-6 carbonate, > 99% enrichment was obtained from Oak Ridge National Laboratory, Isotope Sales Dept., Isotopes Development Center, P. O. Box "X" Oak Ridge, Tenn. 37830.

Attempted Removal of Uranium and Lithium at Four Concentrations of Hydrofluoric Acid

An amount of 20 g. of the Hercules pulp PS-57 was dispersed by hand in 1.0 liter of water; the pulp was collected as a pad on the funnel on one circle of the dry-lapped pulp to restrict the flow of water. Each pad was then leached by percolation with the hydrofluoric acid of the specified concentration followed by 800 ml. of water. Both liquids were poured onto the pad in small portions with intermittent applications of suction from a water aspirator to promote draining. Finally the pads were pressed and dried (Table V).

TABLE V

SAMPLE NUMBERS AND CORRESPONDING STRENGTHS OF ACID

Sample No.	Description
3101-083	2% (<u>1M</u>)
3101-084	0.2% (<u>0.1M</u>)
3101-085	0.02% (<u>0.01M</u>)
3101-086	0.002% (<u>0.001M</u>)
3101-087	Successive leaching with 300 ml. each of 2%, 0.2%, 0.02%, 0.002%, water.

Leaching IPC-1478 Paper with Hydrofluoric Acid

Four 12.5 cm.-circles (6 g.) of IPC-1478 paper (untreated with Kronisol) were sandwiched between two sets of two each of the Hercules PS-57 linter, dry-lapped pulp on the funnel. The stacks of circles were wetted with 100 ml. of water and pressed under a rubber dam to expel air pockets. Hydrofluoric acid (400 ml.) in 100-ml. portions of 0.2%, was percolated through the pad followed by water in small portions until the filtrate was no longer acid to Congo Red paper. The pad was pressed, and dried at 50°. The acid-washed IPC-1478 paper

was designated 3101-092. The experiment was repeated except that 0.02% hydrofluoric acid was used in place of the 0.2% acid. This sample was designated 3101-096.

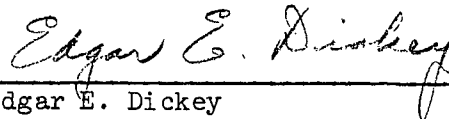
Treatment of Pulp with Ion-Exchange Resins

One pound of the moist mixed-bed resin, Amberlite MB-3 (Mallinckrodt AR) was suspended in sufficient water to occupy a volume of ca. 800 ml. in a 1-liter plastic (Nalgene) beaker. A strip of dry-lapped pulp approximately 6.5 cm. x 40 cm. (15 g.) was placed inside a cellophane dialysis casing (7.4 cm. wide) and sealed on one end by folding and clamping with plastic paper clips. The interior of the dialysis casing was flooded with 0.4% hydrofluoric acid, the air and the excess acid were expelled, and the end was sealed with plastic paper clips, as above. The package was then folded in an accordion fashion, placed in a 1-liter plastic beaker, and covered with the slurry of the mixed-bed resin. After standing at room temperature overnight, the strip of pulp was removed from the casing, was neutral to Congo Red paper, was pressed under a rubber dam, and dried. The sample was designated 3101-089.

LITERATURE CITED

1. Project 3101, Report Three, January 12, 1973.
2. Project 3101, Report Two, November 10, 1972.

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