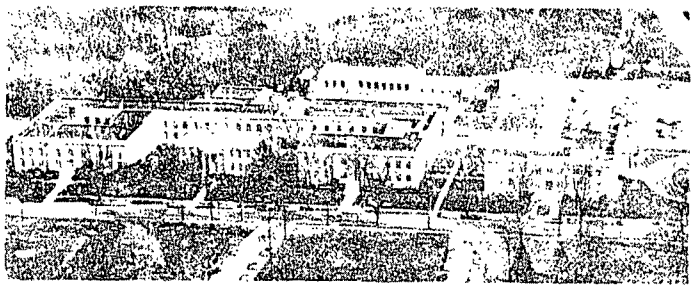

THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

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

Project 3101

Report Seven

Final Report Under Contract No. F08606-72-C-003 as Amended

to

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

August 22, 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

This is Report Seven under IPC Project 3101, the final report for the period July 1, 1972 to June 30, 1973, covered by Contract No. F08606-72-C-003 as amended.

The objective of Project 3101 was to develop a process for the production of a filter paper, with the filtration characteristics equal to those of IPC-1478, and with < 0.3 ng. U/g. of paper with an 8/5 ratio of 137, and < 0.4 ng. Li/g. of the finished paper.

A cotton linters pulp (Hercules, Inc., PS-57) and IPC-1478 filter paper (untreated with Kronisol, dibutoxyethyl phthalate) were treated (leached) at the Institute with several reagents to attempt to reach the specified goal. Complementary experiments were performed at McClellan AFB, MCL-C with samples of the same pulp and with IPC-1478 paper, with and without Kronisol.

Hydrofluoric acid, 0.5M to 2M, and ammonium carbonate, 0.2M to 0.5M, were both effective in lowering the uranium contents of pulp and IPC-1478 paper samples to < 0.3 ng. U/g., with the lowest (Table VII, Sample 138) 0.07 ng. U/g. When used in sequence on the same sample, these reagents appeared to be most efficient.

Both hydrofluoric acid and ammonium carbonate were effective in lowering the lithium contents of the samples, but the acid appears to be more effective than the carbonate. The lowest lithium levels of < 0.15 ng. Li/g. were obtained

for one sample of pulp (Table V, Sample 145) which had been leached, washed, and dried three times in succession, using both ammonium carbonate and hydrofluoric acid.

The most effective technique thus far consisted of percolating the reagents and the wash water under gravity through stacks of circles or disks of pulp or paper.

INTRODUCTION

HISTORICAL

The development of IPC-1478 filter paper was begun in 1948 at The Institute of Paper Chemistry. Between 1950 and the present, the production of the paper evolved through handmade sheets to commercially made paper. A detailed account of the development of IPC-1478 filter paper is contained in a report by Colonel Arnold J. Celick (1).

It was observed early in the development of the paper that the uranium and lithium contents of the finished paper were usually higher than the values for the pulp from which the paper was made (2). This was attributed to the ability of the pulps to sequester cations from the process water. Subsequently, it was found that the presence of ammonium carbonate at pH 9 in the papermaking process depressed the uranium content of finished paper to acceptably low levels (3).

As interest grew in the availability of IPC-1478 paper which was low in both lithium and uranium, data accumulated which indicated that the lithium was loosely bound to the cellulose and that the conditions for lowering the uranium content also favored lowering the lithium content (4). The need to develop filter paper especially low in both uranium and lithium led to the current project at the IPC.

OBJECTIVES OF PROJECT 3101

The objective of Project 3101 was to develop a manufacturing procedure for filter paper with lithium contents < 0.4 ng. Li/g. of paper with a Li^6/Li^7

atom ratio of approximately 0.08 (natural Li), and uranium contents < 0.3 ng. U/g. of paper with a U238/U235 atom ratio approximately 137.5 (natural U). Furthermore, the manufacturing procedure was to be capable of the consistent production of low-lithium, low-uranium filter paper with filtering properties similar to IPC-1478 filter paper (see Appendix I).

The work divided itself roughly into (a) the search for a method to lower the lithium and uranium contents of a suitable pulp to the specified levels followed by the production of filter paper from such pulps, and (b) the leaching of lithium and uranium from intact sheets of filter paper before the treatment with Kronisol[a]. This general approach was used by Gandrud and Lazrus (5) to lower the ash content of IPC-1478 filter paper, except that the paper they used had been treated with Kronisol and water only was used to leach the paper. On Project 3101, both ammonium carbonate and hydrofluoric acid were selected as possible useful reagents to remove the unwanted elements. Ammonium carbonate was selected because of its current usefulness in the commercial production of IPC-1478 paper (1), and hydrofluoric acid was chosen because of its usefulness in the production of "ashless" filter paper (6).

Before the experimental program was begun, E. E. Dickey of the Institute's staff visited the laboratories at McClellan AFB on July 6-7, 1972 and conferred with Captain Frank Grosso and his staff. Background information was reviewed and the objectives of Project 3101 were clarified. On May 22 and 23, 1973, Captains Grosso, Wright, and Paquette, U.S. Air Force, visited the Institute to review the progress of the work and to up-date the plans for future work.

---

[a] Kronisol is dibutoxyethyl phthalate manufactured by the FMC Corp.

DISCUSSION

PROCESS WATER

The removal of uranium and lithium from the pulp and paper under investigation requires that the amounts of these elements in process water be reduced to the lowest possible level. Initially, samples of distilled water (Table I, Samples 007, 017, and MCL) and deionized water were analyzed. As shown in Table I, the deionized samples, especially after additional treatment with a back-up cartridge of mixed-bed resin (008, 098, 135, 136), were practically equal in quality to the distilled water from the IPC Analytical Laboratory and from MCL.

At present the Institute's deionized water (008, 097) is fed to a back-up cartridge of mixed-bed resin, the effluent (098) from which is led into a special high capacity mixed-bed resin in series with an adsorption resin to remove colloidal silica and hydrous metal oxides (099, 135, 136). The special resins were purchased as Research Cartridge No. 1506-30 and Puritan Cartridge No. 1506-40, Cole-Parmer Instrument Co., Chicago, Ill.

Consistent with the generally known behavior of cellulose, mats of pulp purified by extraction with strong acid sequestered some of the trace amounts of uranium and lithium present in the process water. Based on a comparison of Sample 027, Table I with 055, Table II, the use of a closed white water system in the making of paper from purified pulp may be feasible. This would favor the gradual lowering of cations in the pulp to some equilibrium level during the making of a single lot of paper.

Department of the Air Force
1155th Technical Operations Squadron (Hq. Comd.)
McClellan AFB, California
Project 3101

TABLE I

URANIUM AND LITHIUM CONTENTS OF PROCESS WATER[a]

Sample No.	Description	Uranium 8/5	Uranium ng./ml.	Lithium ng./ml
3101-007	Distilled water, IPC Analytical Lab.	136	0.0018	0.0084
008	Deionized water from tap in Room 209, IPC, collected 8-21-72	136	0.00142	0.0090
017	Distilled water, "old still," IPC	137	0.00148	0.0366
009	Tap water, Appleton City water	139	0.052	10.2
026	Deionized water from tap in Room 209, IPC, collected 10-6-72 A (teflon beaker)	129	0.00082	0.79
	B (Pt. dish)	135	0.00056	1.06
027	Double-deionized water; effluent from cartridge of mixed-bed resin fed from deionized tap, Room 209	126	0.00068	0.021
097	Deionized water from tap in Room 209, IPC, collected 1-17-73	113	0.00064	0.0224
098	Effluent from back-up cartridge, collected 1-17-73	97	0.00039	0.0070
099	Effluent from second back-up cartridge and final resin (triple-deionized water), collected 1-17-73	81	0.00039	0.015
135	Triple-deionized water collected 2-8-73	--	--	0.0070
136	Triple-deionized water collected 2-20-73	125	0.00031	0.0058
MCL	Triple-distilled water	--	--	0.010
MCL	Double-distilled water	--	--	0.015

[a]All analytical data were obtained at the McClellan AFB, MCL-C, and were transmitted to the Institute by letter; see Ref. (7).

TABLE II

PULP LEACHED IN SLURRY FORM WITH VARIOUS REAGENTS

Sample No.	Description		Uranium 8/5	Uranium ng./g.	Uranium ng./ml.	Lithium ng./g.	Lithium ng./ml.
3101-001	Control	A	116.6	1.284		2.33	
	Cotton linter pulp,	B	115.6	1.263		2.27	
	Hercules PS-57	C	115.6	1.262		2.27	
		D	115.9	1.248		2.61	
		av.	115.9	1.264		2.37	
002	Pulp washed with distilled water, only	A	130	3.49[a]		3.74	
		B	134	7.09[a]		3.86	
003	Filtrate from 002		128		0.00435		0.073[a]
004	Pulp leached with 0.5M hydrofluoric acid and 0.25M ammonium carbonate	A	137	0.346		2.79	
		B	140	0.453		2.84	
005	Filtrate from hydrofluoric acid treatment of 004		118		0.0274		0.048
006	Filtrate from ammonium carbonate treatment of 004		128		0.0051		0.095
016	Pulp leached with 0.5M sodium fluoride and 0.25M ammonium carbonate	A	138	4.105[a]		4.31	
		B	136	0.897[a]		3.94	
024	Pulp leached with 1.0M hydrofluoric acid and magnesium bicarbonate	A (4)	135[a]	7.97[a]		4.08	
		B (5)	138	0.64		4.89	
		C (6)	137	0.42		2.51	
025	Pulp leached with magnesium bicarbonate, only	A (4)	131	0.61		3.52	
		B (5)	127	0.45		4.44	
		C (6)	128	0.49		3.14	
083	Pulp leached with 1.0M HF	A	129	0.86		0.68	
		B	118	0.34		0.64	
084	Pulp leached with 0.1M HF	A	116	0.32		0.64	
		B	116	0.31		0.63	
085	Pulp leached with 0.01M HF	A	116	0.43		0.51	
		B	116	0.34		0.44	
086	Pulp leached with 0.001M HF	A	118	0.26		0.59	
		B	117	0.21		0.46	
087	Pulp leached with 1.0-0.001M HF	A	116	0.37		0.62	
		B	115	0.38		0.71	
100	Pulp, 100 g., in 4 l. of 0.5M hydrofluoric acid	A	128	0.31		1.35	
		B	137	0.51		1.72	
101	Pulp, 100 g., in 4 l. each of 0.5M HF; NaOH at 2.3 micrograms/g. of pulp; 0.5M HF	A	137	0.47		2.64	
		B	135	1.33		2.91	
053	Water filtrate (2 l.) from the dispersion of 260 g. of pulp in 4 l. of water		59		0.0077		0.20
054	Filtrate (2 l.) from 1.0M hydrofluoric acid leaching of 260 g. pulp (056-058)		115		0.114		0.117
055	Water effluent from purified pulp (056-058)		(36)		0.00053		0.013
056 Top zone	Hercules PS-57 pulp, 260 g., leached with 1.0M hydrofluoric acid; washed with water; dried	A	120	0.29		1.23	
		B	95	0.30		1.19	
057 Middle zone		A	124	0.35		1.04	
		B	118	0.25		0.79	
058 Bottom zone		A	113	0.15		0.56	
		B	113	0.13		0.48	

[a] Average of two analyses.

LEACHING PULP IN SLURRY FORM WITH VARIOUS REAGENTS

Initially, the Hercules pulp was dispersed in water and in solutions of various reagents. After leaching (or soaking) for a specified time, the solution was removed by filtration on a polyethylene Buchner funnel, the mat of pulp was washed with water, pressed under a rubber dam and dried.

Water

As shown in Table II, distilled water apparently added to the uranium and lithium already present (Sample 002). However, the relatively high 8/5 ratio for uranium in the pulp and the somewhat lower ratio in the water washings (Sample 003) suggested that some of the original uranium with an 8/5 ratio of 116 (Control 001) had been exchanged for the uranium in the water. However, no further work was done with water alone.

Hydrofluoric Acid, Ammonium Carbonate, Sodium Fluoride, and Magnesium Bicarbonate

Although Kubinek (6) used a mixture of hydrochloric and hydrofluoric acids in the production of ashless filter paper, the use of dilute hydrofluoric acid, alone, was selected for the early work with the Hercules pulp. Also, ammonium carbonate is used to repress the uranium content in the manufacture of IPC-1478 paper (3). Therefore, both reagents were used with dispersions of the Hercules pulp, and the results are summarized in Table II. The use of 0.5M ammonium carbonate (Sample 004) reduced the uranium to 0.4 ng. U/g. pulp, but the lithium was increased somewhat. The filtrates (005, 006) from the operation were not unusually high in either element.

The use of sodium fluoride (016) and magnesium bicarbonate (024, 025) were deleterious, especially with respect to lithium.

When hydrofluoric acid was used, followed by washing with water only, (Samples 083 to 087, 100), both uranium and lithium were lowered but the levels exceeded those specified (0.3 ng. U and 0.4 ng. Li/g. of pulp). Acid concentration from 0.001M to 1.0M removed, within experimental error, the same amounts of uranium and lithium. This was somewhat unexpected since the higher concentrations might be expected to be more efficient in regenerating the acid form of the cellulose.

The use of a small amount of sodium ion in the hydrofluoric acid to displace the lithium was unsuccessful (Sample 101).

When a large amount (260 g.) of pulp was dispersed in water, formed into a thick pad, and then washed by percolation with hydrofluoric acid followed by water, satisfactory levels of uranium (056-058) were achieved and the bottom section of the pulp mat was nearly acceptable in lithium (058). The final water filtrate (055) from the operation was very low in uranium and lithium due to their possible removal on passing through the pulp pad. This result is consistent with the cation-exchange properties of cellulose, especially after treatment with strong acid.

The percolation of acid through a thick pad of pulp, as described above, involves the countercurrent principle. This was evident in the tendency of both uranium and lithium to drop in the direction of flow of liquid through the pad of pulp. Also, there was somewhat less exchange of the uranium (8/5 ratio of 116) in the pulp with that of the process water (8/5 ratio 137) in the bottom zone (058) of the pad as compared with the top zone (056).

Thus far, efforts to reach the desired levels of uranium and lithium in the cotton linter pulp by leaching as a slurry of 4-5% consistency have been successful only for uranium. Pulps leached with hydrofluoric acid or with acid and ammonium carbonate in sequence retained amounts of lithium in excess of the specified amount. In contrast, the first results of the percolation technique were encouraging, and the technique was applied to stacks of pulp and of IPC-1478 paper. The results are described in subsequent sections of the report.

PERCOLATION TECHNIQUES WITH DRY-LAPPED PULP

Leaching Circles of Pulp with Sodium Fluoride, Ammonium Carbonate, and Hydrofluoric Acid

Circles and disks cut from dry-lapped pulp were arranged in stacks and the reagents and wash water were percolated through the stacks by gravity. This technique, countercurrent in principle, was designed to extract the pulps exhaustively. The results are shown in Table III. The first two experiments (Samples 011 to 015 and 018 to 023) demonstrated the relatively high efficiency for the removal of uranium to levels well below the specified goal, < 0.3 ng. U/g. of pulp. However, the lithium was more tenaciously held at levels well above the required amount, < 0.4 ng. Li/g. Subsequent experiments (115-118) confirmed the general usefulness of the technique. As a further refinement in the technique, the pulp circles were processed and dried between circles of filter paper (118). This provided protection of the pulp circles from air-borne contamination during the drying step and resulted in a lithium content of 0.37 ng./g., essentially the maximum value sought.

TABLE III

PURIFICATION OF PULP BY THE PERCOLATION OF VARIOUS REAGENTS THROUGH STACKS OF DRY-LAPPED PULP

Sample No.	Description		Uranium 8/5	Uranium ng./g.	Uranium ng./ml.	Lithium ng./g.	Lithium ng./ml.
3101-010	Combined sodium fluoride filtrates from 011 to 016		130		0.115		3.25
011 (top)	Stack of pulp circles, 12 cm., Hercules PS-57, cut from dry-lapped sheets; leached with 0.5M sodium fluoride and 0.2M ammonium carbonate		140	5.14		9.23	
012			131	0.18		1.54	
013			130	0.18		1.78	
014			157	0.17		1.89	
015 (bottom)			135	0.61		0.94	
018 (top)		A (1)	137	2.27		11.22	
		B (2)	134	0.15		0.85	
021	Stack of 13 circles of pulp leached with 1.0M hydrofluoric acid; 0.2M ammonium carbonate	A (7)	128	0.11		0.72	
		B (8)	132	0.13		0.71	
		C (9)	131	0.08		1.29	
023		A (12)	136	0.18		0.84	
		B (13)	131	0.13		0.67	
115 (Circles 3 and 4)	110 g. Pulp circles (26 cm.) leached with 0.5M HF containing 23 micrograms of sodium ions/g. pulp; 1 liter 0.025M HF					0.67	
116 (Circles 3 and 4)	110 g. Pulp circles (26 cm.) leached with 0.5M HF containing 23 mg. of sodium; HF at pH 3 containing 23 ng. sodium/g. pulp					1.00	
117 (Circles 5-8)	57.5 g. Pulp circles (12.5 cm.) leached with 0.5M HF containing 13.7 mg. sodium ion; 0.025M HF		119	0.10		0.46	
118 (Circles 5-8)	Same as 117 except circles processed between circles of filter paper					0.37	

Leaching Circles of Pulp with Nitric, Hydrochloric, and Hydrofluoric Acids

As a further test of the percolation technique, circles of pulp were leached with nitric, hydrochloric, and hydrofluoric acids; the results are summarized in Table IV. Except for the somewhat better removal of lithium by hydrofluoric acid than by the other two acids, there is little to be gained by choosing one acid over the others. Although the acids ranged from 0.3M (nitric) to 1.0M (hydrofluoric), no evidence is available that a uniform strength of the three acids would have produced more uniform results. Also, it is recognized that the unique ability of hydrofluoric acid to dissolve siliceous substances may also aid in the removal of cations possibly associated with silicates.

As further evidence of the advantages of the percolation technique, the top circles of each stack of pulp (Table III 011, 018, and Table IV 030, 038, 046) were considerably higher in both uranium and lithium than the remainder of the pulp circles. This was interpreted as (a) the filtering out of particulate contaminations and (b) the exchange of cations for hydrogen ions on the acid (carboxyl) groups of the cellulose.

The reagents and filtrates from the several percolation experiments (Tables III and IV) showed the expected accumulation of uranium and lithium. Furthermore, the 8/5 ratios of 120 to 137 for uranium in the processed pulps clearly indicated that a significant proportion of the uranium (8/5 ratio 116) originally present was exchanged with the more common natural uranium (8/5 ratio 137), as trace impurities in the reagents and the process water.

TABLE IV

COMPARISON OF NITRIC, HYDROCHLORIC, AND HYDROFLUORIC ACIDS IN THE PURIFICATION OF PULP BY PERCOLATION

Sample No.	Description		Uranium 8/5	Uranium ng./g.	Uranium ng./ml.	Lithium ng./g.	Lithium ng./ml.
3101-028	0.3M nitric acid reagent		119		0.0010		0.014
029	Filtrate (1 l.) from 0.3M nitric acid treatment of pulp (030-034)	A (teflon beaker)	115		0.087		0.14
		B (Pt. dish)	115		0.069		0.14
035	Water washings (1 l.) from pulp after 0.3M nitric acid leaching		125		0.0025		0.013
030	Stack of pulp circles (73.7 g.) leached with 0.3M nitric acid; washed with water; dried						
Circle 1 (top)			138	6.50		5.33	
Circle 2			133	0.22		1.58	
032							
Circle 6			123	0.97		0.78	
Circle 7			138	0.11		1.21	
Circle 8			130	0.21		1.15	
034							
Circle 11			135	0.27		0.90	
Circle 12 (bottom)			125	0.12		1.62	
036	0.5M hydrochloric acid reagent		134		0.0011		0.017
037	Filtrate from 0.5M hydrochloric acid treatment of pulp		87		0.072		0.144
043	Water washings from pulp (038-042) after 0.5M hydrochloric acid leaching	A (teflon beaker)	127		0.0018		0.015
		B (Pt. dish)	127		0.0015		0.015
038	Stack of pulp circles (74.0 g.) leached with 0.5M hydrochloric acid; washed with water; dried						
Circle 1 (top)			130	2.10		2.67	
Circle 2			127	0.16		1.00	
040							
Circle 6			126	0.13		1.19	
Circle 7			127	0.14		1.17	
Circle 8			128	0.13		1.01	
042							
Circle 11			125	0.10		1.56	
Circle 12			124	0.10		1.17	
Circle 13 (bottom)			127	0.13		1.19	
044	1.0M hydrofluoric acid reagent		136		0.0036		0.018
045	Filtrate from 1.0M hydrofluoric acid treatment of pulp (046-050)		116		0.139		0.234
051	Water washings from pulp after 1.0M hydrofluoric acid leaching		123		0.0084		0.018
046	Stack of pulp circles (111.1 g.) leached with 1.0M hydrofluoric acid; washed with water; dried						
Circle 1 (top)			136	1.00		2.56	
Circle 2			127	0.21		0.89	
048							
Circle 9			128	0.24		0.69	
Circle 10			85	0.17		0.71	
Circle 11			123	0.16		0.79	
050							
Circle 18			121	0.16		0.59	
Circle 19			120	0.16		0.66	
Circle 20 (bottom)			122	0.20		0.79	

Leaching Disks of Dry-Lapped Pulp with Hydrofluoric Acid and Ammonium Carbonate. Large Scale

Dry-lapped pulp was cut into octagonal disks, 16 cm. in diameter, and stacked in a polyethylene, Buchner funnel. Stacks of 440 to 880 g. of disks were leached by percolation and dried in sets of three. The results are shown in Table V. All samples, except No. 145, were processed through one cycle of leaching, washing, and drying, and retained from 13 to 35% of the lithium originally present. Uranium contents of pulps processed by percolation (Tables III and IV) were known to be < 0.3 ng. U/g., and only selected samples were analyzed for uranium (Table V) in the Large Scale series.

Sample 145 consisted of pulp disks which had been leached, through two cycles, with 0.5M hydrofluoric acid, and then finally with 0.5M ammonium carbonate followed by 2M hydrofluoric acid and water. The uranium content was < 0.1 ng./g. and the lithium < 0.15 ng./g. The repetition of the purification cycle, including the drying step, was indicated as a useful technique to achieve the lithium goal as well as that of uranium.

PULPS TAGGED WITH LITHIUM-6

The difference between the 8/5 ratio (116) for uranium in the Hercules pulp, PS-57, and that (137) of the uranium in reagents and process water was used as a rough indicator for the pattern of exchange and removal of uranium in leaching experiments. In a similar way, pulps were exposed to lithium-6 (> 99% enrichment) in attempts to determine the patterns of lithium removal and exchange. The results are summarized in Table VI. A purified pulp (079) containing 1.1 ng. Li/g. of pulp with a 6/7 ratio of 0.08, was exposed to 4 ng. ^{6}Li/g. of pulp. The labelled pulp (069) contained approximately 4 ng. Li/g., most of which was lithium-6 (6/7

TABLE V

LEACHING DRY-LAPPED PULP WITH HYDROFLUORIC ACID AND AMMONIUM CARBONATE BY PERCOLATION. LARGE SCALE

Sample No.	Description		Uranium 8/5	Uranium ng./g.	Lithium ng./g.
3101-104 (disks 4-7)	869 g. pulp disks (16 cm. octagons) leached with 0.5M HF; water	A	117	0.16	0.55
		B	131	0.51	0.84
107 (disks 34-37)		A	115	0.19	0.69
		B	115	0.18	0.52
110 (disks 64-67)		A	122	0.22	0.49
		B	119	0.23	0.84
113 (disks 94-97)		A	116	0.21	0.63
		B	117	0.21	0.53
119 (upper)	883 g. pulp disks (16 cm. octagons) leached with 0.5M HF; water; then half of stack (440 g.) leached with 0.5M HF; water				0.53
120 (middle)					0.53
121 (lower)					0.55
122 (upper)	500 g. pulp disks (16 cm. octagons) leached with 0.1M ammonium carbonate containing 28 mg. of sodium/g. of pulp; water; 0.5M HF; water				0.77
123 (middle)					0.47
124 (lower)			118	0.19	0.54
125 (upper)	440 g. pulp disks (from 119) leached with 0.5M HF; water				0.41
126 (middle)					0.79
127 (lower)					0.30
145	Pulp disks previously extracted as in Samples 125-127. Then leached with 0.5M ammonium carbonate; water; 2M HF; water	A	136	0.064	0.14[a]
		B	135	0.096	0.11

[a] Represents the average of two analyses.

TABLE VI

PULPS TAGGED WITH LITHIUM-6

Sample No.	Description		Uranium 8/5 ng./g.	Uranium ng./ml.	Lithium 6/7 Main	Lithium 6/7 Spiked	Lithium ng./g.	Lithium ng./ml.
3101-069	Purified pulp (079) exposed to 4 ng./g. of lithium-6	A	120	0.38	2.94	9.13	3.86	
		B	121	0.41	3.46	9.80	4.12	
073	Water filtrate (4 l.) from pulp (258 g.)		115	0.0040	0.0764	3.37		0.086
074	1.0M hydrofluoric acid reagent		133	0.0085	0.0745	0.906		0.341
075	1.0M hydrofluoric acid filtrate (3.6 l.)		118	0.0742	0.0886	0.463		0.765
076	Reagent for lithium-6		92	0.00035	18.72	114.7		0.161
077	Filtrate (3.8 l.) from lithium-6 treatment		114	0.00062	2.653	56.4		0.0060
079	Purified pulp	A	116	0.39	0.0867	6.15	1.11	
		B	122	0.38	0.0784	6.39	1.04	
080	Lithium-6 pulp (069) leached with water	A	129	0.68	0.868	8.73	1.75	
		B	131	1.10	1.430	5.47	3.24	
081	Lithium-6 pulp (069) leached with 1.0M HF; water	A	123	0.068	0.098	7.69	1.03	
		B	131	0.081	0.097	6.19	1.12	
082	1.0M HF filtrate from 20 g. of treated pulp (081, 1 liter)		129	0.0179	0.218	(0.662)		0.712
128 (upper)	400 g. pulp disks (16 cm. octagons) leached with 0.5M HF containing 10 ng. ^{6}Li/g. pulp; 0.5M HF lithium-free; water		115	0.18	0.0828		0.76	
129 (lower)					0.123		0.30	
130 (upper)	400 g. pulp disks (16 cm. octagons) leached with 0.1M ammonium carbonate containing 19 ng. ^{6}Li/g. pulp; 0.1M ammonium carbonate lithium-free; water				0.320		0.55	
131 (lower)					0.543		0.85	

ratio = 3.1). The lithium contents of the water filtrate (073) and the lithium-6 filtrate (077) were about as expected, but the hydrofluoric acid solutions (074, 075) appeared to be abnormally high in lithium. Therefore, no attempt was made to estimate a mass balance for lithium in the system.

When the pulp (069) labelled with lithium-6 was washed with water, some of the lithium was removed, but the uranium content was increased (Sample 080). When the labelled pulp was leached with hydrofluoric acid and washed with water (Sample 081), both the lithium content and the 6/7 ratio returned nearly to the original values. This behavior suggested that (a) some of the original lithium occupied sites which were inaccessible to the lithium-6 reagent and to the hydrofluoric acid or (b) all the lithium was exchanged in the hydrofluoric acid and the final amount was due to absorption from the wash water. The hypotheses were not tested with pulps slurried in solutions of lithium-6, but stacks of pulp disks were leached by percolation. The influence of both hydrofluoric acid and ammonium carbonate on the absorption of lithium-6 was tested. In the presence of hydrofluoric acid, the lithium-6 was retained to only a small degree (Samples 128-129), but the presence of ammonium carbonate appeared to favor the exchange of lithium (Samples 130-131) as indicated by the somewhat higher 6/7 ratios.

Based on the experiments with pulps labelled with lithium-6 most if not all lithium may be exchangeable, particularly if a mildly alkaline reagent, such as ammonium carbonate, is used to leach the pulp (4).

IPC-1478 PAPER LEACHED WITH HYDROFLUORIC ACID AND AMMONIUM CARBONATE

The percolation technique as applied to disks of dry-lapped pulp was applied also to the IPC-1478 paper. The results are summarized in Table VII.

Circles were cut from samples of commercially made paper which had not been treated with Kronisol. This avoided the accumulation of oily residues on the processing equipment and obviated the possible interference of oil films with the removal of lithium and uranium.

In all leaching experiments except one (096) with 0.01M hydrofluoric acid, the uranium was reduced to levels below 0.3 ng. U/g. of pulp. However, in the removal of lithium, the efficiency appeared to be related roughly to the concentration of hydrofluoric acid. At a concentration of 0.01M hydrofluoric acid, the paper retained > 4 ng. Li/g. of pulp (096). At 0.1-0.5M HF the processed paper (092, 102, 103) retained < 3 ng. Li/g., and at 2.0M HF the paper (132, 133, 134, 142, 143, 146) retained 0.6-0.8 ng. Li/g. The use of ammonium carbonate or added sodium ion appeared to have little effect on the removal of lithium (103, 132, 134, 143, 146). The use of lithium-6 (133, 134) indicated that none of the label was retained since the 6/7 ratios of the extracted papers were those of natural lithium. The lithium analysis for Sample 138 appears to have been erratic; however, the uranium analysis indicated that the purification process was very efficient.

Pulp disks (144, 147) which had been used in the processing of the IPC-1478 paper, retained satisfactory levels of both uranium and lithium.

TABLE VII

IPC-1478 PAPER (WITHOUT KRONISOL) LEACHED WITH HYDROFLUORIC ACID AND AMMONIUM CARBONATE

Sample No.	Amount of Paper and MCL Identification	Leaching Reagent		Uranium 8/5	Uranium ng./g.	Lithium 6/7 Unspiked	Lithium ng./g.
013-5501	PM-7061A, Roll 2, Buckeye Linters	Control	A	131	0.800		7.80
			B	137	0.730		7.51
	PM-7062A, Roll 1, Hercules SFP	Control	C	127	1.18		6.44
			D	128	1.26		6.42
	PM-7061A, Roll 1, Buckeye linters, double pass	Control		135	0.95		7.53
3101-001	Pulp, Hercules, PS-57 (see Table II)	Control		116	1.26		2.37
092	Circles	0.1M HF	A	135	0.27		2.78
			B	134	0.27		2.95
096	Circles	0.01M HF	A	137	0.40		4.10
			B	136	0.38		4.15
102	3 Circles, (as above)	0.5M HF; water	A	130	0.27		2.85
			B	129	0.19		2.85
103	3 Circles, (as above)	0.005M ammonium carbonate containing 2.3 mg. of sodium ion; water; 0.5M HF; water	A	136	0.187		2.03
			B	136	0.188		2.92
132	6 Circles, PM-7062B, Roll 1, SFP, single pass	0.1M ammonium carbonate containing 11.5 mg. of sodium ion per g. of paper; water; 2M HF; water		119	0.102		0.692
133	6 Circles, (as in 132)	2M HF containing 10 ng. of ^{6}Li/g. pulp; 2M HF lithium-free; water				0.0812	0.766
134	6 Circles, (as in 132)	0.1M ammonium carbonate containing 11.5 mg. of sodium ion and 10 ng. ^{6}Li/g. paper; water; 2M HF; water				0.0801	0.683
137	Circles, Sample PM-7061A, Roll 2, Buckeye Linter, Double Pass, stacked with disks of Hercules pulp, PS-57	2M hydrofluoric acid; 0.5M hydrofluoric acid; water	A	127	0.148		1.30
			B	126	0.129		0.92
138	Circles, (same as 137)	0.5M ammonium carbonate; water; 2M hydrofluoric acid; water	A	132	0.065		4.12[a]
			B	126	0.074		NR
142	Circles, Sample PM-7062A, Roll 1, Hercules Linters	2M hydrofluoric acid; water		128	0.132		0.666
143	Circles (same as 142)	0.5M ammonium carbonate; water; 2M hydrofluoric acid; water		133	0.165		0.838
144	Pulp disks from stack with 143		A	136	0.223		0.315
			B	134	0.081		0.288
146	Circles, Sample PM-7062A, Roll 1, stacked with previously extracted pulp (145)	0.5M ammonium carbonate; water; 2M hydrofluoric acid; water		132	0.104		0.593[b]
147	Pulp disks from 146		A	141	0.277		0.206[b]
			B	137	0.092		0.186

[a] Average of three analyses.
[b] Average of two analyses.

MISCELLANEOUS EXPERIMENTS

During the course of the work, several experiments were designed to answer specific questions of limited scope. The results are collected in Table VIII.

In one experiment (052) the pulp was leached with hydrofluoric acid, pressed under a rubber dam, and submitted for analysis without drying. The experiment was designed to reveal whether or not the drying step might be a significant source of contamination. The results indicated that the uranium content was acceptable and, according to the 8/5 ratio, was residual from the original pulp (see 001, Table II). The lithium content was approximately the same as that of other pulp samples leached with hydrofluoric acid, washed, and dried in the usual way (079, Table VI; Table III). Therefore, little if any contamination originated with the drying step.

Pulp Leached with a Mixture of Hydrochloric and Hydrofluoric Acids

Pulp was leached with a mixture of hydrochloric and hydrofluoric acids (114) as described by Kubinek (6) for the preparation of "ashless" filter paper. There was no evident advantage of the mixed acids over hydrofluoric acid alone.

Dialysis of Pulp in the Presence of Amberlite MB-3 Ion-Exchange Resin

A strip of the dry-lapped pulp was placed inside a cellophane dialysis casing flooded with 0.2M hydrofluoric acid and the package was then immersed in a slurry of Amberlite MB-3 in water. After standing overnight, the pulp was removed, pressed, and dried. Both uranium and lithium had been reduced to low

TABLE VIII

MISCELLANEOUS EXPERIMENTS

Sample No.	Description		Uranium 8/5	Uranium ng./g.	Lithium ng./g.
3101-052	Circles of pulp leached with 1.0M hydrofluoric acid followed by 0.01M hydrofluoric acid; not dried before shipment; dried at MCL	A	110	0.26	0.90
		B	91	0.28	1.01
		C	115	3.58	1.12
114	100 g. of pulp dispersed in 4 liters of 2% of a mixture of HCl:HF (2:1)	A	132	0.45	2.44
		B	125	0.18	0.81
089	Pulp + 0.2M hydrofluoric acid in dialysis casing; dialyzed against Amberlite MB-3	A	123	0.36	0.64
		B	127	0.42	0.68
139 (upper section)	Pulp disks, Hercules PS-57, previously leached; Stack of pulp disks and paper; leached with 2M hydrofluoric acid; water	A	116	0.19	0.38
		B	120	0.21	0.37
140	Pulp, reduced with sodium borohydride; Stack of pulp disks and paper; leached with 2M hydrofluoric acid; water		135	1.55	0.31
141 (lower section)	Pulp disks (same as 139); Stack of pulp disks and paper; leached with 2M hydrofluoric acid; water	A	118	0.34	0.42
		B	118	0.33	0.35
067	Blotter stock, control	A	140	17.6	--[a]
		B	138	17.0	--[a]
060	1 (top), 2; Blotter stock (59.8 g.) leached with 1.0M hydrofluoric acid; water; dried		137	0.787	2.00 (top)
062	Circle 6; Blotter stock (59.8 g.) leached with 1.0M hydrofluoric acid; water; dried	A	137	0.59	11.5
	Circle 7; Blotter stock (59.8 g.) leached with 1.0M hydrofluoric acid; water; dried	B	137	0.63	1.35
	Circle 8; Blotter stock (59.8 g.) leached with 1.0M hydrofluoric acid; water; dried	C	138	0.58	--
063	Circle 9, Circle 10, Circle 11; Blotter stock (59.8 g.) leached with 1.0M hydrofluoric acid; water; dried		138	0.60	1.36
066	Circle 19, Circle 20, Circle 21 (bottom); Blotter stock (59.8 g.) leached with 1.0M hydrofluoric acid; water; dried		139	0.64	--[a]
059	2% HF filtrate (1 liter) from blotter stock		138	1.10 ng./ml.	1.00 ng./ml.

[a] Lithium analyses were unsuccessful for these samples.

levels but were in excess of the specifications. No work is planned to explore this method further.

Treatment of Pulp with Sodium Borohydride

A small amount of pulp was treated with sodium borohydride to attempt to diminish the carbonyl groups possibly present. Subsequent leaching of the pulp with 2M hydrofluoric acid was designed to remove the sodium and other impurities possibly introduced by the reduction process. The results (140) showed that the process held no advantage over those involving only hydrofluoric acid and/or ammonium carbonate.

Pulp circles, which had been extracted previously, were placed above and below the mat of borohydride-treated pulp to shield it from impurities. These samples (139, 141) were essentially below the levels sought for uranium and lithium.

Blotter Stock (for Handsheet-Making) Leached with Hydrofluoric Acid

It was known from previous work that blotters used at the IPC for couching handsheets were quite high in uranium and lithium as compared with the cotton linters pulp, Hercules PS-57. In order to test the capabilites of the percolation procedure, circles of the blotter stock were leached with hydrofluoric acid, washed with water, and dried. The results (059 to 067) indicated a high efficiency toward the removal of uranium even though the amounts were considerably above the specified amount. The data for lithium are erratic but tend to show, as did the uranium data, that the percolation method may be useful in the purification of such blotter stock. This may be desirable in the preparation of handsheets as planned for the extension of the project.

PULP AND PAPER SAMPLES PROCESSED AT McCLELLAN AFB, MCL-C

Data for samples of pulp and IPC-1478 paper (with and without Kronisol) were supplied by McClellan AFB, MCL-C, for comparative purposes. The samples were processed and analyzed in the laboratories of MCL from which the experimental details are available.

As shown in Table IX, when the Hercules pulp, PS-57, was leached in slurry form with either hydrochloric acid at pH 3 (012-5535) or ammonium carbonate at pH 9 (012-5536), both uranium and lithium values were greatly improved. The treatment with ammonium carbonate produced uranium values within the specified limit.

The percolation procedure was applied to stacks of circles cut from IPC-1478 paper, with and without Kronisol (012-5539, -5540, -5541). The best results reported to date (012-5541) were obtained by leaching the paper with ammonium carbonate followed by hydrochloric acid. Although the uranium values were within that specified (< 0.3 ng. U/g.), the lithium levels were 1.8-2.8 ng. Li/g. or approximately half the original amount. The specified limit is < 0.4 ng. Li/g. of pulp.

The results obtained at MCL-C are generally similar to those obtained on Institute Project 3101. The fact that very dilute reagents were used at MCL-C suggests that the removal of lithium from IPC-1478 paper may require the higher concentrations of reagents as used by IPC.

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1155th Technical Operations Squadron (Hq. Comd.)
McClellan AFB, California
Project 3101

TABLE IX

PULP AND PAPER SAMPLES PROCESSED AT McCLELLAN AFB, MCL-C

MCL Sample No.	Description		Uranium 8/5	Uranium ng./g.	Lithium ng./g.
--	Pulp control, IPC No. 3101-001		116	1.26	2.37
012-5535	Pulp leached with hydrochloric acid at pH 3	A	118	0.55	0.42
		B	119	0.57	0.89
		C	117	0.56	0.55
		D	117	0.57	0.64
012-5536	Pulp leached with ammonium carbonate at pH 9	A	118	0.23	0.92
		B	120	0.14	0.92
		C	119	0.21	0.59
		D	121	0.24	0.52
012-5537	Stack of circles of IPC-1478 (control) filter paper, with Kronisol, leached with hydrochloric acid at pH 3 and ammonium carbonate at pH 9	A	126	0.32	4.68
		B	136	0.35	2.67
		C	132	0.27	(6.35)
		D	131	0.27	3.32
		E	132	0.30	3.00
		F	133	0.30	2.85
		G	134	0.35	3.51
		H	132	0.34	2.12
		I	132	0.26	2.04
		J	130	0.23	1.42
		K	133	0.22	1.78
--	IPC-1478 paper, control, June, 1970				7.53
012-5538	IPC-1478 paper, control, November, 1972	A	133	0.83	8.62
		B	134	0.88	8.95
012-5539	Paper without Kronisol; 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	126	0.36	4.71
		B	(99)	(0.61)	(7.96)
		C	107	0.33	4.32
		D	109	0.35	5.00
		E	126	0.33	4.32
		F	(55)	0.41	5.36
		G	117	0.36	4.93
		H	106	0.34	5.44
		I	125	0.37	4.82
		J	124	0.38	4.74
012-5540	Paper without Kronisol; 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	130	0.42	4.80
		C	133	0.45	4.60
		D	133	0.52	4.87
		E	134	0.65	5.87
		F	126	0.46	5.12
		G	132	0.51	5.10
		H	133	0.52	5.50
		I	131	0.48	4.02
		J	133	0.46	4.18
012-5541	Paper with Kronisol				
	Control	A	123	0.40	5.26
	Control	B	(71)	0.80	5.96
	Stack of sixteen circles leached with ammonium carbonate pH 9, water-washed, leached with hydrochloric acid at pH 3, water-washed, dried	C	117	0.29	3.67
		D	127	0.27	2.63
		E	124	0.16	2.90
		F	127	0.17	1.99
		G	127	0.24	2.07
		H	121	0.19	2.25
		I	107	0.17	2.13
		J	128	0.23	5.98
		K	127	0.16	1.84
		L	87	0.19	2.20
		M	122	0.14	2.11
		N	128	0.19	1.98
		P	129	0.25	1.80
		R	129	0.23	2.33
		S	133	0.26	2.90
		T	129	0.24	2.56

EXPERIMENTAL

ANALYSIS OF SAMPLES

The analysis of pulp, water, and reagent samples for lithium and uranium was done under the direction of Captain Frank Grosso and, in part, under Captain James Wright, McClellan AFB, MCL-C (7). The analytical data in this report are uncorrected for blanks.

Samples were assigned unique numbers in the Series 3101-001 to 3101-999,[a] as suggested by Captain Grosso, and were sent prepaid by United Parcel Service to McClellan AFB, MCL-C.

001[a] Cotton Linters Pulp

Through the efforts of Captain Frank Grosso, McClellan AFB, MCL-C, arrangements were made to secure a supply of a purified cotton linters pulp from a lot reserved for McClellan AFB by Hercules, Inc. As instructed by W. J. Molaison, Hercules Incorporated, Wilmington, Delaware, we placed an order on July 13 for one bale (400 lb.) with Hercules Incorporated, C and SP Dept., 120 Oakbrook Central Mall, Suite 5000, Oak Brook, Illinois 60521. The pulp was designated by Hercules as PS-57 Cotton Linters, OSR No. 92868, and was furnished as dry-lapped sheets with a basis weight of 93.5 lb. of airdry pulp per 1000 sq. ft. The pulp was delivered on August 3, 1972. The wrapping had been damaged in shipment and on two faces of the bale the exposed pulp was soiled. The soiled pulp was removed on a small paper trimmer and the pulp was repackaged in polyethylene sheeting in small packages, 20-30 lb. each.

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[a]All sample numbers assigned by the IPC are composed of a four-digit number 3101, the same as the Project number, followed by a unique three-digit number. In this section the four-digit project number is omitted for the sake of simplicity.

IPC-1478 Filter Paper

All samples of IPC-1478 filter paper, processed at the Institute, were obtained from McClellan AFB, MCL-C, and were not treated with Kronisol.

002, 003 Water-Washed Pulp

An amount of 20 g. (airdry) of the cotton linters pulp as received was dispersed by hand in 500 ml. of distilled water in a 1-liter "Nalgene" beaker with a "Nalgene" stirring rod. After 90 minutes with occasional stirring by hand, the pulp was collected on a 12-cm. polyethylene Buchner funnel (this filtrate was assigned 003), was washed on the funnel with 200 ml. of water, and the wet pulp was divided (by weight) into four equal parts. Each part was dispersed in 500 ml. of distilled water, the mixture was poured onto the 12-cm. funnel, was formed into a pad which was pressed with a clean rubber dam under a water pump vacuum, and the pad was dried at 50° in an oven with gravity circulation of air. The dry pads were stored in polyethylene bags.

004-006 Treatment with Hydrofluoric Acid and Ammonium Carbonate

Hercules cotton linters, 20 g., was dispersed by hand in 500 ml. of distilled water, collected on the funnel under a water pump vacuum, and then leached in 500 ml. of 0.5M hydrofluoric acid (Mallinckrodt AR diluted with distilled water) with occasional stirring over a period of 90 minutes. The pulp was again collected on the funnel (this filtrate was assigned 005), the pad was washed with five 100-ml. portions of 0.5M hydrofluoric acid followed by five 100-ml. portions of distilled water. The wet pad was then leached for 90 minutes in 500 ml. of 0.25M ammonium carbonate (Mallinckrodt AR) with occasional stirring, was recovered on the funnel (this filtrate was assigned 006), washed

with 500 ml. of 0.25M ammonium carbonate followed by 500 ml. of water. The wet pulp was divided into four equal parts and made into pads (see above).

007-009 Water Samples

Deionized water is distilled from all-glass equipment and stored in polyethylene for use in the IPC Analytical Laboratories (007). Deionized water from the tap in Room 209, IPC was designated 008. Tap water, Appleton City water was designated 009.

010-015 Treatment of Intact Sheets of Dry-Lapped Pulp with Sodium Fluoride and Ammonium Carbonate

Five circles 12 cm. in diameter (26.6 g.) were cut from sheets of the Hercules pulp as received and were placed on the septum of the polyethylene funnel. Sodium fluoride (Mallinckrodt AR), 500 ml. of 0.5M, was percolated through the pad in portions under gravity over 60 min. Then followed in succession 200 ml. of distilled water, 500 ml. of 0.25M ammonium carbonate, and 200 ml. of distilled water. Finally, the pad was pressed with the rubber dam under a water pump vacuum, the circles were separated and numbered from 1 to 5, top to bottom, and dried at 50°. The samples were designated 011-015.

The sodium fluoride filtrates from 011-015 and 016 (see next experiment) were combined as 010.

016 Treatment of Pulp with Sodium Fluoride and Ammonium Carbonate

The experiment described under 004-006 was repeated except that 0.5M sodium fluoride was used in place of hydrofluoric acid.

017 Distilled Water (Old Still)

018-023 Treatment of Intact Sheets of Pulp with Hydrofluoric Acid and Ammonium Carbonate

Thirteen circles (75.7 g.) cut from the Hercules Pulp (see above) were packed on the septum of the funnel and the pad was leached with 250 ml. of water. Hydrofluoric acid, 1.0 liter of 1.0M, was percolated through the pad over a period of 30 min. This was followed in succession with 500 ml. of water, 1.0 liter of 0.25M ammonium carbonate, and 500 ml. of water. The pad was pressed with a rubber dam under a water pump vacuum, the circles were separated and numbered from top to bottom, and dried at 50°.

024 Treatment of Pulp with Hydrofluoric Acid and Magnesium Bicarbonate

Magnesium carbonate (Mallinckrodt AR), 30 g., was placed in a half-gal. polyethylene bottle with 1.5 liters of water and the mixture was saturated with carbon dioxide over a period of 60 minutes. The mixture was allowed to stand overnight and the clear supernatant solution of magnesium bicarbonate (approximately 1% expressed as magnesium carbonate) was used in the treatment of pulps.

Hercules pulp, 30 g., was dispersed by hand in 750 ml. of 1.0M hydrofluoric acid and was allowed to stand with occasional stirring at room temperature for 90 min. The pulp was collected on the funnel, washed with 250 ml. of 1.0M hydrofluoric acid, and 500 ml. of distilled water. The last few milliliters were mildly acidic to Congo Red paper. The wet pad was then dispersed in 750 ml. of saturated magnesium bicarbonate (see below). After occasional stirring over a period of 90 min., the pulp was collected on the funnel, was washed with 500 ml. of distilled water, and pressed with a rubber dam under a water pump vacuum. The wet pulp was divided into six equal parts by weight. The first three parts (no. 1, 2, 3) were made into pads and dried in the usual way. The other three parts

(no. 4, 5, 6) were made into pads, each one in succession using the filtrate ("white water") from the preceding pad.

025 Treatment of Pulp with Magnesium Bicarbonate

Hercules pulp, 30 g., was dispersed by hand in 750 ml. of saturated magnesium bicarbonate and stirred occasionally by hand over a period of 90 min. The pulp was collected on the funnel, washed with 250 ml. of the magnesium bicarbonate solution and with 500 ml. of distilled water, and then was pressed free of liquid water under a rubber dam. The wet pulp was then made into six pads (as described above).

026-027 Deionized Water Samples

028-035 Treatment of Pulp with Nitric Acid

A stack of 12 circles (73.7 g.) of Hercules PS-57 pulp was packed on a polyethylene Buchner funnel and 1.0 liter of 0.3M nitric acid (20.0 ml. of concentrated nitric acid diluted to 1.0 liter) was percolated through the stack under gravity. The acid was added in several portions over a period of 60-90 minutes. The stack was washed with 1.0 liter of water, pressed by a rubber dam under a partial vacuum (water aspirator), the circles were separated, numbered, and air-dried at room temperature under a current of filtered air. The reagent and filtrates were sampled, and the circles of pulp were grouped and numbered for analysis.

Sample No.	Description
3101-028	0.3M Nitric acid reagent
029	0.3M Nitric acid filtrate from the pulp (030, etc.)
035	Water washings after the nitric acid treatment
030	Circles 1 (top), 2
031[a]	Circles 3, 4, 5
032	Circles 6, 7, 8
033[a]	Circles 9, 10
034	Circles 11, 12 (bottom)

[a]Not analyzed; retained by IPC.

036-043 Treatment of Pulp with Hydrochloric Acid

A stack of 13 circles (74.0 g.) of Hercules PS-57 pulp was leached by percolation with 1 liter of 0.5M hydrochloric acid followed by 1 liter of water according to the general procedure described for the nitric acid purification (see above). The following samples were collected.

Sample No.	Description
3101-036	0.5M Hydrochloric acid reagent
037	0.5M Hydrochloric acid filtrate from the pulp (038, etc.)
043	Water washings after the hydrochloric acid treatment
038	Circles 1 (top), 2

Sample No.	Description
3101-039[a]	Circles 3, 4, 5
040	Circles 6, 7, 8
041[a]	Circles 9, 10
042	Circles 11, 12, 13 (bottom)

[a]Not analyzed; retained by IPC.

044-051 Treatment of Pulp with Hydrofluoric Acid

A stack of 20 circles (111.1 g.) of Hercules PS-57 pulp was leached by percolation with 1 liter of 1.0M hydrofluoric acid followed by 1.5 l. of water according to the general procedure described for the nitric acid purification (see above). The following samples were collected.

Sample No.	Description
3101-044	1.0M Hydrofluoric acid reagent
045	1.0M Hydrofluoric acid filtrate from the pulp (046, etc.)
051	Water washings from the hydrofluoric acid treatment
046	Circles 1 (top), 2
047[a]	Circles 3, 4, 5, 6, 7, 8
048	Circles 9, 10, 11
049[a]	Circles 12, 13, 14, 15, 16, 17
050	Circles 18, 19, 20 (bottom)

[a]Not analyzed; retained by IPC.

052 Treatment of Pulp with Hydrofluoric Acid, Without Washing or Drying the Pulp

A stack of 6 circles of Hercules PS-57 pulp was leached with 500 ml. of 1.0M hydrofluoric acid followed by 500 ml. of 0.025M hydrofluoric acid (strongly acid to Congo Red). The pad was then pressed under a rubber dam in the usual way. Without drying, the third, fourth, and fifth circles were packaged as a single sample in polyethylene and were submitted for analysis.

053-058 Treatment of Pulp with Hydrofluoric Acid. Large Batch

A large polyethylene bottle was fashioned into a percolation tube, 14.5 cm. in diameter and 20 cm. in height. The bottom was trimmed, perforated with 1/16 inch holes, and placed in the inverted bottle as a septum. Hercules pulp, PS-57, was dispersed in water and poured into the tube in two 80 gram-lots and two 50 gram-lots for a total of 260 g. of airdry pulp. A total of 4 l. of water was used for the dispersion of the pulp. This involved the reuse of the filtrate as each lot was dispersed and added to the column. The charge of pulp formed a loosely compacted column 13 cm. in depth. After the final addition of pulp, 2 l. of filtrate was collected and numbered 053. Two liters of 1.0M hydrofluoric acid was then percolated through pulp and 2 l. of acid filtrate was collected and numbered 054. The pulp bed was then washed with water until free of acid (Congo Red paper), after which a sample was collected for analysis, 055. A total of 4.5 l. of water was required following the acid in order to collect 1 liter of acid-free effluent. Pulp was removed from the top, middle, and bottom zones of the column of pulp, pressed under a rubber dam, and dried in an oven at 50° under gravity circulation of air.

Sample No.	Description
3101-053	Water filtrate (2 l.) from the dispersion of 260 g. of pulp
054	1.0M Hydrofluoric acid filtrate (2 l.) from 260 g. pulp
055	Acid-free water effluent from the purified pulp
056	Top zone
057	Middle zone
058	Bottom zone

059-067 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 1.0M 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 (059). The circles were washed, pressed, and dried in the usual way (Table IV).

Sample No.	Description
3101-059	1.0M 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

Sample No.	Description
3101-064[a]	Circles 12, 13, 14, 15
065[a]	Circles 16, 17, 18
066	Circles 19, 20, 21 (bottom)
067	Original blotter stock, control

[a]Not submitted for analysis; retained by IPC.

068-079 Pulp Labelled 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 (see 053-058 above) 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 073. The mat (column) of pulp was leached by percolation under gravity with 2 liters of 1.0M 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 079, and (3) 184 g. The 1.0M hydrofluoric acid reagent was designated 074, and the combined acid filtrate and washings, 3.6 liters, was designated 075.

Treatment with Lithium-6

The third portion of purified pulp, 184 g., was dispersed in 3 liters of water containing 736 ng. of lithium-6 in the form of lithium-6 carbonate, > 99% enrichment[a]. After 90 minutes with occasional stirring by hand, the pulp was

[a]Lithium-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.

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 069, and served as the lithium-6 control pulp. A portion of the lithium-6 reagent was designated 076, and the aqueous filtrate, 3.8 liters, from treating the pulp, 077.

080-082 Attempted Removal of Lithium-6 from Lithium-6 Treated Pulp

Leaching with Water

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

Leaching with Hydrofluoric Acid

An amount of 10 g. of the ^{6}Li-treated pulp (069) was leached with 250 ml. of 1.0M hydrofluoric acid for 30 min., the slurry was filtered, the pulp was washed with 300 ml. of water, pressed and dried as Sample 081. The combined filtrate and washings from 20 g. of ^{6}Li-pulp leached with acid was designated 082.

083-087 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 X).

TABLE X

SAMPLE NUMBERS AND CORRESPONDING STRENGTHS OF HYDROFLUORIC ACID

Sample No.	Description
3101-083	1M
084	0.1M
085	0.01M
086	0.001M
087	Successive leaching with 300 ml. each of 1.0, 0.1, 0.01, 0.001M

088-089 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 0.2M 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.

090-096 Leaching IPC-1478 Paper with Hydrofluoric Acid

Four 12.5 cm.-circles (6 g.) of IPC-1478 paper (untreated with Kronisol) were sandwiched on the funnel between two sets of two each of the Hercules PS-57 linters, dry-lapped pulp. 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 0.1M, 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 pulp circles were designated 090 and 091, and the IPC-1478 paper was designated 092. The experiment was repeated except that 0.01M hydrofluoric acid was used in place of the 0.1M acid. The pulp circles were designated 094 and 095, and the sample was designated 096.

097-099 Process Water

Samples of deionized water were collected and submitted for analysis (see Table I).

100 Pulp Leached with 0.5M Hydrofluoric Acid

Airdry pulp, 100 g. was dispersed in 4 liters of 0.5M hydrofluoric acid in a Nalgene (polypropylene) beaker under mechanical stirring with a stainless steel stirrer. When the dispersion was complete, the stirrer was removed and the mixture was allowed to stand for sixty minutes. The pulp was formed into a mat on a polyethylene table top Buchner funnel, 26.5 cm. in diameter, the mat was washed with 3 liters of water, pressed under a rubber dam, and dried at 50°C. in an oven with gravity-circulation of air.

101 Attempted Exchange of Sodium for Lithium in Pulp

An amount of 100 g. of pulp was leached with 0.5M hydrofluoric acid and washed with water as described in 100 above. The wet pulp was then dispersed in 4 liters of sodium hydroxide containing 2300 ng. Na/g. of pulp, the mixture was allowed to stand for sixty minutes, was collected on the funnel without washing, and was pressed and dried overnight. The pulp was then leached with 0.5M hydrofluoric acid, washed with water, pressed, and dried as with 100.

102 Leaching IPC-1478 Paper with Hydrofluoric Acid

Three circles of IPC-1478 paper (12.5 cm. in diameter) were placed on top one circle of pulp in a polyethylene funnel and three circles of pulp were placed on top the IPC-1478 paper. An amount of 1.0 liter of 0.5M hydrofluoric acid was percolated through the stack of circles, followed by 1.0 liter of water. The stack of circles was pressed under a rubber dam and the three paper circles were dried together in contact with the contacting pulp circle (third from top).

103 Leaching IPC-1478 Paper with Ammonium Carbonate (with Sodium Ions) and Hydrofluoric Acid

A stack of IPC-1478 paper and of pulp circles was arranged as in 102, and was leached by percolation with 1.0 liter of a solution containing 5 g. of ammonium carbonate and 2.3 mg. of sodium ion (as hydroxide). The stack was then washed with 500 ml. of water followed by 1.0 liter of 0.5M hydrofluoric acid, and finally with 1.0 liter of water. The sample was air-dried as in 102.

104-113 Large-Scale Leaching of Pulp Disks with Hydrofluoric Acid

A quantity of the Hercules PS-57 in the dry-lapped form was cut into regular octagons (16 cm. in diameter) on a guillotine paper cutter. A stack of 103 disks (869 g.) was placed on the septum of the 26.5 cm. table top Buchner funnel, a weight of 5 kg. was placed on top the stack to prevent buckling and the formation of air pockets. Four liters of 0.5M hydrofluoric acid was percolated through the stack which swelled from a height of 9 cm. to 15 cm. during the process. Four liters of water were used to wash the stack at which point the effluent was neutral to Congo Red paper. The stack was separated into pairs of disks for drying and samples for analysis (104, 107, 110, and 113) each consisted of four disks from the top, bottom, and two intermediate zones of the stack. Samples 105, 106, 108, 109, 111, and 112 were stored for possible future use.

114 Pulp Leached with a Mixture of Hydrochloric and Hydrofluoric Acids

Following the general procedure of Kubinek (6), 4 liters of a mixture containing hydrochloric (168 ml. of 38% HCl) and hydrofluoric (34.4 ml. of 50% HF) acids was prepared. Pulp, 100 g., was dispersed in the acid under mechanical stirring, the pulp was collected on the funnel, washed with 5 liters of water, pressed, and dried.

115 Pulp (26 cm.) Leached with Hydrofluoric Acid Containing Sodium Fluoride

Five circles (26 cm. diameter) (110 g.) were cut from dry-lapped pulp and placed in the large funnel (26.5 cm. diameter), and 2 liters of 0.5M hydrofluoric acid containing 23 micrograms of sodium ion per gram of pulp was percolated through the stack of pulp. The stack was washed with 1 liter of 0.025M hydrofluoric acid followed by water until the effluent was neutral to Congo Red paper, pressed under a rubber dam, and dried in the usual way. The 3rd and 4th circles were selected as the sample for analysis.

The experiment described under 115 was repeated except that the pulp was leached with 2 liters of 0.5M hydrofluoric acid containing 25 mg. of sodium ion (110 ml. of 0.01N sodium hydroxide). Also, the pulp was washed with 1 liter of 0.025M hydrofluoric acid followed by 2 liters of hydrofluoric acid at pH 3 containing 23 ng. Na per gram of pulp. The sample was designated 116.

In a similar experiment ten circles, 12.5 cm. in diameter, of pulp were stacked in the small funnel and were leached by percolation with 1 liter of 0.5M hydrofluoric acid containing 12 mg. of sodium ion (55 ml. of 0.01N sodium hydroxide). The stack was then washed with 0.5 liter of 0.025M hydrofluoric acid followed

by 0.5 liter of water, and pressed and dried. Circles 5-8 were submitted as a single sample for analysis as 117.

The above experiment was repeated except that circles of qualitative filter paper were placed above and below each pulp circle from 5 to 8. Thus, these pulp circles were dried between circles of qualitative filter paper to shield them from air-borne contamination. The sample was designated 118.

119 to 121 Pulp Disks Leached with Hydrofluoric Acid, Double Treatment

A stack of pulp disks, 883 g. of 16 cm. octagons, was leached by percolation with 2.0 liters of 0.5M hydrofluoric acid followed by 2.0 liters of water. The stack was then divided into two equal parts, the upper part was pressed, the disks were air-dried, and were used in the preparation of Samples 125 to 127. The lower half of the stack was leached by percolation with 1.0 liter of 0.5M hydrofluoric acid followed by water until the effluent was neutral to Congo Red paper. The stack of disks was pressed, separated into pairs for drying, and sampled as upper, middle, and bottom sections, 119-121, respectively.

122 to 124 Pulp Disks Leached with Ammonium Carbonate (with Sodium Ions) and Hydrofluoric Acid

A stack of pulp disks, 500 g., was leached by percolation with 1.5 liters of 0.1M ammonium carbonate containing 28 micrograms of sodium ion per gram of pulp. The stack was then washed with 1.9 liters of water followed by 1.5 liters of 0.5M hydrofluoric acid, and finally with 2.0 liters of water. The stack of disks was separated into sets of three and only the middle disk of each set was used in analytical samples: upper, middle, and lower sections of the stack, 122, 123, and 124, respectively.

125 to 127 Leaching Previously Treated Pulp Disks with Hydrofluoric Acid

An amount of 440 g. of previously treated disks of pulp (see 119 procedure above) was leached with 1.25 liters of 0.5M hydrofluoric acid, washed with 1.75 liters of water, and dried in sets of three as described under 122. The analytical samples were designated: upper, middle, and lower, 125, 126, and 127, respectively.

128-129 Pulp Disks Leached with Hydrofluoric Acid Containing Lithium-6

A stack of pulp disks, 400 g., was leached with 0.5 liter of 0.5M hydrofluoric acid containing 10 ng. ^{6}Li/g. pulp. This was followed by 0.5 liter of 0.5M hydrofluoric acid which contained no added lithium, and the stack was washed with 1.5 liters of water. The stack was pressed, and the disks were dried in sets of three with samples for analysis from the upper and lower sections of the stack.

130-131 Pulp Disks Leached with Ammonium Carbonate Containing Lithium-6

A stack of pulp disks, 400 g., was leached with 0.9 liter of 0.1M ammonium carbonate containing 19 ng. ^{6}Li/g. pulp. The stack was then leached with 0.5 liter of 0.1M ammonium carbonate containing no added lithium, followed by 1.5 liters of water, pressed, and dried in sets of three. Samples of the upper and lower sections of the stack were analyzed.

132 IPC-1478 Paper Leached with Ammonium Carbonate and Hydrofluoric Acid

Six circles, 12.5 cm. in diameter, of IPC-1478 paper (PM 7062B, Roll 1, SFP, single pass) were placed in sets of two between qualitative filter paper circles. These units were stacked on three circles of pulp and three circles of pulp were placed on top. The stack, total weight 49 g., was leached with

0.5 liter of 0.1M ammonium carbonate containing 11.5 micrograms of sodium ion per gram of fiber. The stack was then washed with 0.5 liter of water, leached with 0.5 liter of 2M hydrofluoric acid followed by 0.7 liter of water. The IPC-1478 paper was dried in sets of two with the qualitative filter paper in place to shield the paper from air-borne contamination.

133 IPC-1478 Paper Leached with Hydrofluoric Acid Containing Lithium-6

Six circles of IPC-1478 paper were arranged in a stack with pulp and qualitative filter paper as described under 132. The stack was leached with 0.5 liter of 2M hydrofluoric acid containing 10 ng. $^{6}Li/g.$ of fiber in the stack, was washed free of acid with water, pressed, and dried.

134 IPC-Paper Leached with Ammonium Carbonate Containing Sodium and Lithium-6, and with Hydrofluoric Acid

Six circles of IPC-1478 paper were packed in the funnel as described under 132; total weight of the stack, 50 g. The stack was leached with 0.5 liter of 0.1M ammonium carbonate containing 11.5 micrograms of sodium ion and 10 ng. lithium-6 per gram of fiber. The carbonate solution was followed by 0.5 liter of water, then 0.5 liter of 2M hydrofluoric acid and finally with 0.7 liter of water. The paper was pressed and dried as described under 132.

137 IPC-1478 Paper Leached with Hydrofluoric Acid

Four circles of IPC-1478 paper, 12.5 cm. in diameter [PM 7061A, Roll 2, Buckeye Linters, Double Pass] were stacked with disks (octagons 15 cm. in diameter) so that each circle of paper was sandwiched between pulp disks. Five additional pulp disks were placed on top and two on the bottom of the stack. The stack was weighted and leached by gravity with 0.6 liter of 2M hydrofluoric acid then 0.8 liter of 0.5M hydrofluoric acid, and finally with 1.2 liters of

water. Each solution was applied in small portions so that the exposure of the stack to each step was about 60 minutes. The stack was pressed and the circles were dried between pulps disks.

138 IPC-1478 Paper Leached with Ammonium Carbonate and Hydrofluoric Acid

Six circles of the IPC-1478 paper [same lot as in 137] were sandwiched between pulp disks as previously described. The stack was leached with 1.0 liter of 0.5M ammonium carbonate, 0.8 liter of water, 0.7 liter of 2M hydrofluoric acid, and finally with 1.5 liters of water. The stack was in contact with each solution for sixty minutes, the final water-wash was completed after standing overnight, and the stack was pressed and dried.

139-141 Pulp Treated with Sodium Borohydride and Leached with Hydrofluoric Acid

An amount of 10 g. of pulp which had been treated with sodium borohydride was suspended in water and formed into a mat 12.5 cm. in diameter. The mat was placed between the 15th and 16th disks in a stack of 22 disks of pulp. The stack was weighted, leached with 0.7 liter of 2M hydrofluoric acid followed by 0.5 liter 0.5M hydrofluoric acid, and finally with water until the effluent was neutral to Congo Red paper. The stack was pressed under a rubber dam, the disks 11-14, and the treated mat, 17-20 were submitted for analysis as 139, 140, and 141, respectively.

142 to 144 IPC-1478 Paper Leached with Hydrofluoric Acid and Ammonium Carbonate

Four circles (12.5 cm.) of IPC-1478 paper [PM 7062A, Roll 1, Hercules[a] Linters] in sets of two each were placed in a stack of previously processed (119)

[a]The package in which this sample was received from McClellan AFB, MCL-C, was labeled "Buckeye," but the paper is believed to have been made from a Hercules pulp.

disks of pulp. The stack consisted of 22 disks (200 g.) with one set of the IPC-1478 paper placed between the 16th and 17th disks and the other set between the 18th and 19th disks. The stack was weighted and was leached with 0.6 liter of 2M hydrofluoric acid followed by 0.7 liter of water. The stack was pressed in the usual way and dried as No. 142.

The experiment was repeated except that the stack was leached with 0.5 liter of 0.5M ammonium carbonate and water before the extraction with hydrofluoric acid. The sample was dried as No. 143. Three sets of three each of the pulp disks stacked above the IPC-1478 paper were dried and the middle disks of each set were combined as pulp Sample 144.

145 Pulp Leached Repeatedly with Hydrofluoric Acid and Ammonium Carbonate

An amount of 200 g. of pulp disks had been leached with 0.5M hydrofluoric acid, washed with water, and dried (see 125 to 127). The stack was weighted and was leached with 0.5 liter of 0.5M ammonium carbonate, washed with 0.7 liter of water, leached with 0.5 liter of 2M hydrofluoric acid, washed with water (1.5 liters) until neutral to Congo Red paper, and dried in sets of three. The middle disks were collected for analysis.

146-147 IPC-1478 Paper Leached with Ammonium Carbonate and Hydrofluoric Acid

Six circles (12.5 cm. in diameter) of IPC-1478 paper [PM 7062A, Roll 1] were placed in sets of two each in a stack of pulp disks (same as 145) which had previously been extracted. The stack was then leached with 0.5 liter of 0.5M ammonium carbonate, 0.5 liter of water, 1.0 liter of 2M hydrofluoric acid, 1.5 liters of water, pressed, and dried. The IPC-1478 paper (146) was dried between

pulp disks. Four pulp disks from the section of the stack above the IPC-1478 paper were submitted as 147.

LITERATURE CITED

1. Celick, Colonel Arnold J. Technical evolution of IPC Filter Paper MCL-TM-70-73, August 20, 1970.

2. Reference (1), page 20.

3. Reference (1), page 35-7.

4. Reference (1), page 37-8.

5. Gandrud, B. W., and Lazrus, A. L., Environmental Sci. Technol. 6:455(1972).

6. Kubinek, V., Papir a celulosa 12:170-3(1957); CA 52:14166a.

7. All analytical data were obtained at the McClellan AFB, MCL-C, and were transmitted to E. E. Dickey from Captain Frank Grosso in letters dated September 22, October 31, 1972, and June 15, 1973; and from Captain James Wright in letters dated December 21, 1972, February 5 and 20, March 12, and April 5, 1973.

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APPENDIX I

STATEMENT OF WORK TO BE DONE ON PROJECT 3101 (AFTAC PROJECT AUTHORIZATION NO. T/2608/L/ETR)

1. Objective: The objective of this project is to develop a manufacturing procedure for low-lithium, low-uranium content filter paper having filtering properties similar to IPC-1478 filter paper.

2. Tasks:

   a. The contractor will perform a research and development project with the intent of developing a procedure to manufacture a filter paper with the following properties:

      (1) Lithium content not to exceed 0.4 ng. Li/g. of paper with a Li6/Li7 atom ratio approximately equal to 0.08 (natural value).

      (2) Uranium content not to exceed 0.3 ng. Li/g. of paper with a U238/U235 atom ratio approximately equal to 137.5 (natural value). The manufacturing procedure should be such that filter paper samples can be produced routinely with similar low-lithium and low-uranium content.

   b. The contractor should give first consideration to the use of cotton linters in developing this procedure. If this goal is unattainable, the contractor should investigate synthetic fibers (e.g., polystyrene).

   c. Throughout his investigations, the contractor shall submit filter paper, processed linter, fiber and/or liquid solution samples (e.g., reagents, soaking solution) to the McClellan Central Laboratory (MCL), for uranium and lithium analysis.

      (1) Total filter paper area for samples should be at least 50 square inches. This will allow sufficient samples for duplicate analysis.

      (2) To avoid contamination by handling, all filter paper samples should be handled with nontalc rubber or plastic gloves and shipped in special plastic bags supplied by MCL.

      (3) All sample bags should be individually numbered and the contractor should include a brief description of the procedures used to obtain each sample batch.

      (4) All liquid solutions should be shipped in clean polyethylene bottles and contain approximately 1 liter of solution. Bottles should be labeled.

d. The contractor will submit progress reports every two months throughout the duration of the project. A personal visit by the contractor may be substituted for a progress report; however, this substitution should be used no more than twice during the project. A comprehensive final report must be submitted within three months after completion of work. The report should detail the final manufacturing procedures and the experiments performed during the work. Negative results must be reported.

APPENDIX II

IPC PROJECT 3101 SAMPLES LISTED IN CONSECUTIVE ORDER
LOCATION OF ANALYTICAL DATA BY TABLE

Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.
001	I, VII[a]	021	III	041	--[a]	061	--[a]	081	VI	101	II[a]	121	V[a]	141	VIII
2	II[a]	022	--[a]	042	IV	062	VIII	082	VI	102	VII	122	V	142	VII
3	II	023	III	043	IV	063	VIII	083	II[a]	103	VII	123	V	143	VII
4	II[a]	024	II[a]	044	IV	064	--[a]	084	II[a]	104	V	124	V	144	VII
5	II	025	II[a]	045	IV	065	--[a]	085	II[a]	105	--[a]	125	V	145	V
6	II	026	I	046	IV	066	VIII	086	II[a]	106	--[a]	126	V	146	VII
7	I	027	I	047	--[a]	067	VIII	087	II	107	V	127	V	147	VII
8	I	028	IV	048	IV	068	--[a]	088	--[a]	108	--[a]	128	VI		
9	I	029	IV	049	--[a]	069	VI	089	VIII	109	--[a]	129	VI		
10	III	030	IV	050	IV	070	--[a]	090	--[a]	110	V	130	VI		
11	III	031	--[a]	051	IV	071	--[a]	091	--[a]	111	--[a]	131	VI		
12	III	032	IV	052	VIII	072	--[a]	092	VII	112	--[a]	132	VII		
13	III	033	--[a]	053	II	073	VI	093	--[a]	113	V	133	VII		
14	III	034	IV	054	II	074	VI	094	--[a]	114	VIII[a]	134	VII		
15	III	035	IV	055	II	075	VI	095	--[a]	115	III	135	I		
16	II[a]	036	IV	056	II	076	VI	096	VII	116	III	136	I		
17	I	037	IV	057	II	077	VI	097	I	117	III	137	VII		
18	III	038	IV	058	II	078	--[a]	098	I	118	III	138	VII		
19	--[a]	039	--[a]	059	VIII	079	VI	099	I	119	V[a]	139	VIII		
20	--[a]	040	IV	060	VIII	080	VI	100	II[a]	120	V[a]	140	VIII		

[a] See Appendix III for list of surplus samples.

APPENDIX III

LIST OF SURPLUS SAMPLES OF PULP AND IPC-1478 PAPER FROM PROJECT 3101 ON HAND AT THE INSTITUTE OF PAPER CHEMISTRY

Sample No.	Amount, g.	Analysis Available, Table No.	Description
3101-001	--	II, VII	Any sample of the Hercules linter pulp, PS-57
002	5	II	Water-washed pulp
004	5	II	Pulp leached with 0.5M HF and 0.25M ammonium carbonate
016	5	II	Pulp leached with 0.5M NaF and 0.25M ammonium carbonate
019	10	--	Circles 3, 4 — Pulp circles (12 cm. diameter) leached with 1.0M HF and 0.25M ammonium carbonate [Anal. similar to 021 and 023, Table III]
020	10	--	Circles 5, 6 — Pulp circles (12 cm. diameter) leached with 1.0M HF and 0.25M ammonium carbonate [Anal. similar to 021 and 023, Table III]
022	10	--	Circles 10, 11 — Pulp circles (12 cm. diameter) leached with 1.0M HF and 0.25M ammonium carbonate [Anal. similar to 021 and 023, Table III]
024	15	II	Pulp leached with 1.0M HF and magnesium bicarbonate
025	15	II	Pulp leached with magnesium bicarbonate, only
031	15	--	Circles 3, 4, 5 — Pulp leached with 0.3M HNO_3 [Anal. similar to 032 and 034, Table IV]
033	10	--	Circles 9, 10 — Pulp leached with 0.3M HNO_3 [Anal. similar to 032 and 034, Table IV]
039	15	--	Circles 3, 4, 5 — Pulp leached with 0.5M [Anal. similar to 040 and 042, Table IV]
041	10	--	Circles 9, 10 — Pulp leached with 0.5M [Anal. similar to 040 and 042, Table IV]
047	30	--	Circles 3-8 — Pulp leached with 1.0M HF [Anal. similar to 048 and 050, Table IV]
049	30	--	Circles 12-17 — Pulp leached with 1.0M HF [Anal. similar to 048 and 050, Table IV]
061	10	--	Circles 3-5 — Blotter stock leached with 1.0M [Anal. similar to 063 and 066, Table VIII]
064	12	--	Circles 12-15 — Blotter stock leached with 1.0M [Anal. similar to 063 and 066, Table VIII]
065	10	--	Circles 16-18 — Blotter stock leached with 1.0M [Anal. similar to 063 and 066, Table VIII]
068	23	--	Purified pulp labeled with lithium-6 [Anal. similar to 069, Table VI]
070	22	--	Purified pulp labeled with lithium-6 [Anal. similar to 069, Table VI]
071	28	--	Purified pulp labeled with lithium-6 [Anal. similar to 069, Table VI]
072	23	--	Purified pulp labeled with lithium-6 [Anal. similar to 069, Table VI]
078	50	--	Pulp leached with 1.0M HF, top section [Anal. similar to 079]
083	20	II	Pulp leached with 1.0M HF
084	20	II	Pulp leached with 0.1M HF
085	20	II	Pulp leached with 0.01M HF
086	20	II	Pulp leached with 0.001M HF
088	5	--	Pulp deionized with ion-exchange resin [Anal. similar to 089, Table VIII]

Department of the Air Force
1155th Technical Operations Squadron (Hq. Comd.)
McClellan AFB, California
Project 3101

APPENDIX III (Continued)

LIST OF SURPLUS SAMPLES OF PULP AND IPC-1478 PAPER FROM PROJECT 3101 ON HAND AT THE INSTITUTE OF PAPER CHEMISTRY

Sample No.	Amount, g.	Analysis Available, Table No.	Description
090	5	--	Circle 1 (top) — Pulp circles from leaching of IPC-1478 paper with 0.1M HF [see 092, Table VII]
091	5	--	Circle 2 — Pulp circles from leaching of IPC-1478 paper with 0.1M HF [see 092, Table VII]
093	5	--	IPC-1478 paper scraps leached with 0.1M HF
094	5	--	Circle 1 (top) — Pulp circles from leaching of IPC-1478 paper with 0.01M HF [see 096, Table VII]
095	5	--	Circle 2 — Pulp circles from leaching of IPC-1478 paper with 0.01M HF [see 096, Table VII]
100	50	II	Pulp leached with 0.5M HF
101	40	II	Pulp leached with 0.5M HF containing NaF
105	30	--	Disks 14-17 — Pulp disks leached with 0.5M HF [Anal. similar to 104 to 113, Table V]
106	30	--	Disks 24-27 — Pulp disks leached with 0.5M HF [Anal. similar to 104 to 113, Table V]
108	30	--	Disks 44-47 — Pulp disks leached with 0.5M HF [Anal. similar to 104 to 113, Table V]
109	30	--	Disks 54-57 — Pulp disks leached with 0.5M HF [Anal. similar to 104 to 113, Table V]
111	30	--	Disks 74-77 — Pulp disks leached with 0.5M HF [Anal. similar to 104 to 113, Table V]
112	30	--	Disks 84-87 — Pulp disks leached with 0.5M HF [Anal. similar to 104 to 113, Table V]
114	10	VIII	Pulp leached with 2% of mixture of HCl and HF
119	30	V	Top section — Pulp disks (16 cm. octagons) leached with 0.5M HF
120	30	V	Middle section — Pulp disks (16 cm. octagons) leached with 0.5M HF
121	30	V	Bottom section — Pulp disks (16 cm. octagons) leached with 0.5M HF