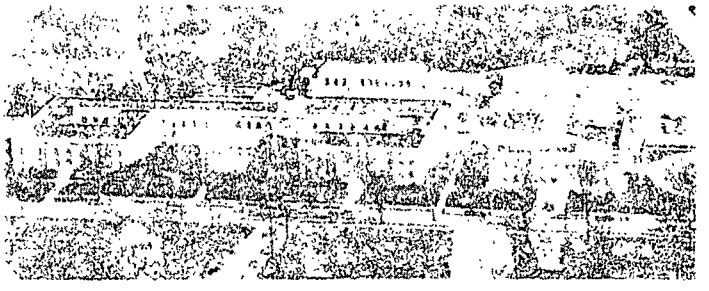

THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

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

Project 3101-1

Report Nineteen

Final Report Under Contract No. F08608-75-C-0009 as Amended

to

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

July 7, 1975

THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

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THE INSTITUTE OF PAPER CHEMISTRY

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

SUMMARY

This is Report Nineteen under IPC Project 3101-1, the final report for the period July 1, 1974 to June 30, 1975, covered by Contract No. F08608-75-C-0009. The previous two year's contracts in this series were covered by Reports Seven and Fourteen.

Two processes are described in the Conclusions by which low-uranium, low-lithium content filter paper may be made. Consistent levels of uranium and lithium were <0.1 ng U/g and 1-2 ng Li/g of paper, respectively. The level of uranium is well within the specification of <0.3 ng U/g, but the lithium exceeds the specification of <0.4 ng Li/g set by the U.S. Air Force for this contract. At present, no practical process is available for the production of IPC-1478 paper at <1.0 ng Li/g.

A process of leaching IPC-1478 paper, Series N, in 19-inch squares with 0.1M hydrofluoric acid and 0.1M ammonium carbonate, followed by thorough washing with deionized water was applied to thirteen 100-sheet lots of paper. Kronisol was applied at the rate of 30% based on the airdry paper. The uranium contents of the finished paper were generally <0.2 ng U/g and those of lithium were 1-2 ng Li/g.

The general procedure of leaching paper with chemical agents and water was applied successfully to intact rolls of Series N paper.

Purification of Series N paper and Hercules pulp by leaching with chemical agents and then exhaustive washing with deionized water was unsuccessful in lowering the lithium below the 1-2 ng/g level.

Fines were shown to retain as much as 10 times the concentration of lithium as the accompanying fibers. However, the relatively small proportion of fines contributed a minor fraction of the lithium in IPC-1478 paper. Furthermore, the purification process was equally effective in removing the lithium from the fines and the fibers.

Controlled leaching of Series N paper with water demonstrated that >80% of the lithium was readily accessible to purified water.

Series N paper was somewhat less efficiently purified in a capillary movement technique than in percolation techniques. Glass equipment did not appear to supply lithium to the system.

The use of reagents and water at 80°C was without effect in comparison with reagents and water at room temperature.

A series of sets of handsheets was made in tap water (Appleton city water, pH 9.6; 11.1 ng Li/ml) from Hercules PS-57 pulp and from purified pulp. The results demonstrated that approximately 90% of the lithium in the newly formed, wet sheet may be washed out with purified water applied before couching the sheet. There was some added advantage in using wash water at pH 9 (ammonium carbonate), and in the use of purified pulp in purified water to form the sheets.

Selected samples of pulp and paper were analyzed by emission spectrography. The purification procedure generally removed 85-91% of the ash (by muffle

furnace at 550°C). The removal of sodium was 84-94%, similar to that of lithium as determined by mass spectrometry at McClellan AFB-MCL-C. The removal of manganese was similar in efficiency to that of uranium. Thus, sodium and manganese may be considered as indicators for lithium and uranium, respectively, in the purification process.

Air filtration tests on selected samples were run at Knowlton Brothers in the USAF tester under the supervision of Dr. Grant Rauscher. The results showed that the purification processes for IPC-1478 paper caused little if any changes in the filtration properties of the paper.

Included herewith is a copy of a memorandum on Project 3101-1 concerning the pilot-plant tests conducted at Knowlton Brothers, Watertown, NY, in August, 1974.

Department of the Air Force
1155th Technical Operations Squadron (Hq. Comd.)
McClellan AFB, California
Project 3101-1

INTRODUCTION

PROJECT 3101 — THE FIRST YEAR

Between July 1, 1972 and June 30, 1973, a program of research resulted in a general laboratory method for the purification of pulp and IPC-1478 paper. Among the methods tested the most effective consisted of leaching stacks of pulp and/or paper under gravity with dilute aqueous solutions of acids and bases, washing with deionized water, and drying at room temperature in a stream of filtered air. The most efficient reagents were hydrofluoric acid and ammonium carbonate. The best results were obtained with a purified cotton linters pulp (Hercules, Inc., PS-57) which retained only 0.07 ng U/g and <0.15 ng Li/g.

The details of the first year's work were consolidated as Report Seven under Project 3101, a final report covered by Contract No. F08606-72-C-003.

PROJECT 3101 — THE SECOND YEAR

Between July 1, 1973 and June 30, 1974 the purification procedures were refined and extended with emphasis on the removal of lithium from commercially-made IPC-1478 paper, and the use of purified pulp in handmade paper. The best results were obtained for handsheets made (without scrim) in all-plastic equipment from purified pulp. These handsheets retained approximately 0.6 ng Li/g and supported the premise that IPC-1478 paper might be made from purified pulp, with lithium levels approaching the specified goal of <0.4 ng Li/g.

A few small samples of commercially-made IPC-1478 paper were available and were leached in stacks with sheets of dry-lapped pulp, PS-57. Although the

pulp samples consistently retained less lithium than the paper, the purification process was effective in producing levels of 0.5-0.7 ng Li/g in the IPC-1478 paper.

IPC-1478 paper, Series N, made at Knowlton Brothers, Watertown, NY, in October, 1973, was unusually high in lithium, 20 ng Li/g. Through the application of the purification process to stacks of this paper followed by the application of Kronisol, lithium levels of 1-2 ng Li/g were achieved. The remainder of the lithium resisted all efforts to reach the specified goal.

Chemical agents other than hydrofluoric acid and ammonium carbonate were tested in the purification procedure, but none possessed any advantage over those reagents. The others included ammonium bicarbonate, tetramethyl ammonium hydroxide, ammonium hydroxide, sodium hydroxide, ammonium fluoride, ammonium chloride, ethyl alcohol, oxalic acid, and EDTA (disodium ethylenediamine tetraacetate).

The details of the second year's work included discussions of possible sources of lithium in IPC-1478 paper and of the production of high purity water. All details were consolidated as Report Fourteen under Project 3101, a final report covered by Contract No. F08606-74-C-0016.

PROJECT 3101-1* — THE THIRD YEAR

This report covers the work on Project 3101-1 for the period between July 1, 1974 and June 30, 1975, and is Report Nineteen, the final report for the work covered by Contract No. F08608-75-C-0009 as amended. The reader may

*The "dash one" (-1) was added by the Institute's Business Office for accounting purposes.

consult Project 3101, Reports Seven and Fourteen, for the details of work accomplished under Contract No. F08606-72-C-003 and F08606-74-C-0016, respectively. Only essential details from Reports Seven and Fourteen are repeated in the current report (Report Nineteen).

Under Conclusions, this report, p. 27, two processes are outlined for the production of low-uranium, low-lithium paper.

DISCUSSION

DEIONIZED WATER

Deionized water was used throughout the work on Project 3101-1 except where unpurified tap water was specified in the study of the retention of lithium in handsheets (393, 397). Usually the lithium levels (Table I) of the deionized water were <0.01 ng Li/ml and contributed <0.05 ng Li/g to the final lithium content of the finished pulp and paper samples. Reports Seven and Fourteen may be consulted for more data on possible relationships between the lithium carried in the water and that retained in purified paper (1,2).

PURIFICATION OF IPC-1478 PAPER, SERIES N

In line with the objective of Project 3101-1, work was continued to develop a process for the production of low-uranium and low-lithium paper. During the current contract period, July 1, 1974 to June 30, 1975, considerable effort was spent in testing the efficiency and precision of a method which had evolved during the first two years' work (1,2). This involved the processing of twelve lots of Series N paper averaging approximately 100 sheets/lot. The analytical data for the purified paper are summarized in Table II.

Sample 365 (Table II) represented the first application of the purification procedure to a relatively large amount of paper. The stack of 35 sheets (20-inch* squares) was cut from Series N paper, Roll No. 22. The purification

*(Roll No. 22 was 40 inches wide, which afforded 20-inch squares. All other rolls of Series N paper were 38 inches wide, which resulted in 19-inch squares in order to use the paper efficiently.

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TABLE I

DEIONIZED PROCESS WATER FROM ROOM 1229, IPC

Sample No. 3101-	Uranium 8/5	Uranium ng/g	Lithium, ng/g	Date Collected
367	122	0.0006	0.022	September 17, 1974
369			0.008	September 23, 1974
371	125	0.0009	0.006	September 30, 1974
373			0.009	October 7, 1974
375	122	0.0005	0.004	October 14, 1974
377			0.005	October 21, 1974
379	121	0.0006	0.011	November 4, 1974
382			0.010	November 11, 1974
395	124	0.004	0.009	December 5, 1974
408			0.007	December 17, 1974
411	143	0.0009	0.009[a]	January 8, 1975
Note: A fresh cartridge of deionizing resin was placed on stream January 9, 1975				
424	138	0.0005	0.007	January 13, 1975
471	134	0.0012	0.013	March 10, 1975
478	133	0.0019	0.017	April 7, 1975
487	133	0.0008	0.0046	April 28, 1975
390	138	0.149	11.1	Appleton city water; collected Dec. 3, 1974

[a]The first values for lithium were 0.62 and 0.67 ng Li/ml but were rejected due to apparent errors in the analysis.

TABLE II

PURIFICATION OF IPC-1478 PAPER, SERIES N, IN 19 AND 20-INCH SQUARES

Lot No.	Sample No. 3101-		Uranium δ/5	Uranium ng/g	Lithium, ng/g	Remarks
--	365-1		133	0.24	5.10	35 Sheets, 20-inch squares from Roll No. 22.
	-2		130	0.14	2.49	Dry ash of these samples was gray-white in
	-3		123	0.53	4.95	color. Kronisol applied by capillary migration
	392		137	0.0095	1.29	1 Liter of Kronisol
1	366-1		133	0.11	5.42	120 Sheets from Roll No. 11-8; one purification cycle. 30% Kronisol was applied from a paint sprayer. Every tenth sheet was selected for analysis. Dry ash of these samples was brown in color
	-2		137	0.21	3.69	
	-3		138	0.20	3.70	
	-4		141	0.20	3.95	
	-5		135	0.19	3.83	
	-6		122	0.12	6.71	
	-7		137	0.092	5.66	
	-8		128	0.090	5.16	
	-9		140	0.089	5.79	
	-10		133	0.088	7.22	
	-11		138	0.095	7.74	
	-12		(105)	(0.15)	8.52	
2	368				1.43	1st Cycle
	376	A	131	0.119	3.87	2nd Cycle, 30% Kronisol added with paint sprayer — from Roll No. 11-9
		B	130	0.071	2.71	
				(calc.	1.40)	
3	370		125	0.068	1.34	1st Cycle
	378	A	130	0.205	2.60	2nd Cycle, 30% Kronisol added with paint sprayer — from Roll No. 11-7
		B	129	0.084	2.08	
				(calc.	1.33)	
4	372	A	143	0.149	1.36	1st Cycle, 5 hours
		B	132	0.126	1.54	
	406		120	0.148	1.16	2nd Cycle, 5 hours — from Roll No. 11-4
	409		140	0.122	1.48	30% Kronisol added with plastic applicator
5	374		137	0.137	2.78[a]	1st Cycle, 5 hours
	410		135	0.154	1.20	2nd Cycle, 5 hours — from Roll No. 11-1
	425		138	0.148	1.19	30% Kronisol added
6	386		141	0.118	1.57	1st Cycle, 5 hours
	423		126	0.064	0.94	2nd Cycle, 8 days — from Roll No. 11-5
	426		141	0.062	1.11	30% Kronisol added
7	389		133	0.100	1.81	1st Cycle, 5 hours
	427		131	0.091	1.54	2nd Cycle, 5 hours — from Roll No. 11-2
	428		135	0.096	1.19	30% Kronisol added
8	398		134	0.126	2.33	1st Cycle, 5 hours
	429		122	0.083	1.59	2nd Cycle, 8 days — from Roll No. 11-3
	430		131	0.058	1.23	30% Kronisol added
9	470		132	0.139	2.98	1st Cycle, 5 hours
	472		135	0.107	1.72	2nd Cycle, 5 hours — from Roll No. 1-3
	473		134	0.109	--	30% Kronisol added
10	474		135	0.089	1.84	1st Cycle, 5 hours
	475		136	0.139	1.20	2nd Cycle, 3 days — from Roll No. 1-5
	476		136	0.093	1.75	30% Kronisol added
11	477		129	0.139	2.16	1st Cycle, 5 hours — 200 Sheets from Rolls No. 1-6; 1-15, combined
	483		134	0.192	1.12	2nd Cycle, 5 hours
	484		136	0.102	2.20	30% Kronisol added
12	485		133	0.093	1.94	1st Cycle, 5 hours
	486		134	0.135	1.41	2nd Cycle, 3 days — From Roll No. 11-10
	488		132	0.044	1.48	30% Kronisol added

[a]The sample was reanalyzed as 374C. The first two values were 40 and 44 ng Li/g and were believed to be in error.

procedure was somewhat ineffective, and the levels of uranium and lithium resembled those of Series L paper.

The next attempt was made with 120 sheets (19-inch squares) of Series N paper. The purification was similar to that of Sample 365, but the Kronisol was applied with a small paint sprayer rather than by capillary migration. The analysis of every tenth sheet (Table II, Sample 366) showed a low level of uranium with a trend toward lower values from top to bottom of the stack. The 8/5 ratio for the uranium resembled more closely the 8/5 ratio (approximately 130) of the process water (see Table I) than that (118) of the original linter pulp. As in many previous experiments the uranium levels were well below the specified objective of <0.3 ng U/g.

On the other hand, lithium was retained at levels of 3.7 to 8.5 ng Li/g and showed a trend toward higher levels from top to bottom of the stack, the reverse of the trend for uranium. No further work was done to explore the observed trends in the distribution of uranium and lithium in purified paper, but such phenomena may be helpful in optimizing purification procedures.

The remainder of the 19-inch squares was purified by similar procedures all of which included two cycles of leaching and drying. Also, the purification procedure was modified to extend the time of contact of the paper with the reagents and wash water (Samples 423, 429, 475, 486), but no significant improvement was observed in comparison with samples of short exposure times (Samples 410, 427, 472, 483).

A small paint sprayer was used to apply the Kronisol to Samples 366, 368, and 370 and was suspected but not established as a source of lithium contamination.

Even so, a simple applicator was made from a 500-ml PE (polyethylene) bottle for the application of Kronisol. The data for Lots 4-12 (Table II) indicate that the application of Kronisol added no lithium to the finished paper in addition to that (1.29 ng Li/g) carried by the Kronisol itself.

The analyses of the finished paper were consistent from Lots 4-12 with uranium <0.1 ng U/g and lithium <1.8 ng Li/g.

PURIFICATION OF SERIES N PAPER IN INTACT ROLLS

Rolls of Series N paper were provided by the laboratory at McClellan AFB. Each roll was 10 inches by 80 feet and, at the Institute, was wound on an inert core of 3-1/2-inch PVC sewer pipe. The rolls were leached in two cycles by the percolation of hydrofluoric acid and ammonium carbonate, thoroughly washed with water, pressed and dried. The data listed in Table III for Samples 383 and 384 show that the purification method may be satisfactory for intact rolls. However, a dryer with a drape-fold rack is necessary to dry the intact sheet.

LIMITED LEACHING OF SERIES N PAPER

In order to explore the accessibility of lithium, a stack of 100 sheets of Series N paper was leached with deionized water by percolation and capillary action. The water was added carefully in small portions until the stack was just wet. The stack was separated into sets of four sheets each, and dried in a stream of filtered air. The data in Table III, Samples 385A to Y, show that >80% of the lithium was removed from the top set and was carried progressively to the bottom set in a relatively high concentration, >8 times the original (20 ng Li/g).

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TABLE III

ANALYSIS OF SAMPLES FROM MISCELLANEOUS EXPERIMENTS[a]

Sample No. 3101-	Weight Analyzed, g	Uranium 8/5	Uranium ng/g	Lithium, ng/g	Remarks
IPC-1478 paper, Series N, purified in the roll by leaching with 0.1M hydrofluoric acid and 0.1M ammonium carbonate.					
383		133	0.143	1.85	1st Cycle, Roll No. 2
384		141	0.107	1.29	2nd Cycle
IPC-1478 paper, Series N, 17-cm squares, stack of 100, leached by percolation with 1300 ml of deionized water; dried and packaged in sets of four.					
385 A				3.32	Sheets 1-4 [Note: The remainder of this series is available for analysis as surplus samples.]
F				8.56	Sheets 21-24
L				17.3	Sheets 45-48
U				44.2	Sheets 81-84
Y				167	Sheets 97-100
IPC-1478 paper, Series N, strips 24-cm wide by 110-cm long, leached by capillary migration of water and ammonium carbonate.					
399				2.52	In PE trough; water only
401				2.87	In PE trough; water and amm. carbonate
402				2.35	In glass trough; water and amm. carbonate
Hercules pulp, PS-57, and IPC-1478 paper, Series N, were leached with 0.1M hydrofluoric acid and 0.1M ammonium carbonate and washed with water. The liquids were transported through a wick by capillarity.					
438	8.65	135	0.287	0.909	PS-57 pulp, top, adjacent to Series N paper
439	6.53	137	0.175	1.06	Series N paper
441		136	0.121	1.22	
443		136	0.212	1.03	
444		133	0.133	0.76	PS-57 pulp, bottom, adjacent to the Series N pulp
Hercules pulp, PS-57 (262 g) was purified by leaching with 0.1M hydrofluoric acid and 0.1M ammonium carbonate, and was washed exhaustively with water. Samples were selected for analysis from the midsection and the lower section of the mat.					
448	7.71	135	0.096	3.40	Midsection
451	9.48	137	0.240	0.51	Lower section
453	5.823	127	1.63	3.00	Wt. of fines, 0.354 g; Fines from fiber Samples 445-452 (262 g pulp)
454	5.540	129	1.27	2.11	Wt. of fines, 0.152 g

Most of the lithium appeared to be available in a water-soluble form and was readily washed from the fiber. The distribution from top to bottom, 385A to Y, appeared to be consistent with the phenomenon of diffusion as encountered in the washing of chemical pulps.

ATTEMPTED PURIFICATION OF SERIES N PAPER BY CAPILLARY MIGRATION OF THE LEACHING AGENTS

In order to test the efficiency of lithium removal when the leaching agents moved through the sheet by capillary migration, strips of Series N paper were placed in an apparatus commonly used for paper chromatography. One trough was polyethylene (PE) and neither the paper nor the leaching agents came into contact with glass. The other trough was glass as supplied with such equipment. As shown in Table III, Samples 399, 401, and 402, the efficiency was high but somewhat less than that of other procedures (compare with Samples 406, 410, and 423 in Table II). It seems evident that the all-glass apparatus was not a source of lithium contamination although such equipment could not be used for the hydrofluoric acid treatment.

In another experiment Hercules pulp and Series N paper were leached with reagents and wash water transported through a wick by capillarity. The washing step was extended over several days and involved unusually large amounts of water. As shown in Table III, Samples 438-444, the amount of uranium was somewhat higher than in others of the purified samples (example: Table II). The 8/5 ratio resembled that of the process water more closely than that of the original pulp (8/5 ratio 116-120), and indicated an essentially complete exchange of the uranium in the fiber with that in the process water. The levels of lithium were somewhat higher than usually found for purified pulp but the

levels in the Series N paper were within experimental error of those in other samples (Table II).

PURIFICATION OF HERCULES PULP BY PERCOLATION AND EXHAUSTIVE WASHING. COLLECTION OF FINES*

Hercules pulp, PS-57, was dispersed in water, formed into a thick mat, leached by percolation with hydrofluoric acid and ammonium carbonate, and washed exhaustively over a period of several days. In the dispersion process some of the fines were dislodged and were not recombined with the fibers (see below). The results in Table III for two representative analytical samples (448 and 451) of the mat showed considerable scatter and no evidence that exhaustive washing was of any advantage in the purification process.

The fines dislodged in the dispersion of the pulp were collected on tared circles of purified pulp. As listed in Table III, Samples 453 and 454, the levels of uranium and lithium in the fines were considerably higher than the average of the original pulp. These observations are consistent with others regarding the persistence of lithium in pulp fines (3).

HANDSHEETS MADE IN TAP WATER AND IN DEIONIZED WATER

Both Hercules cotton linter pulp, PS-57, and purified pulp No. 341 (3) were made into handsheets using tap water (Appleton city water) at pH 9 as delivered in the water mains. The lithium content was 11 ng/ml (390, Table I). As shown in Table IV the handsheets, which were dried at approximately 25%

*"Fines" are defined here arbitrarily as particles which will pass through a coarse mesh (50-60) screen but are collected on a suitable fiber mat. The particles may be very short and broken pieces of fibers along with unknown materials which have accumulated during the processing of the pulp and paper.

TABLE IV

HANDSHEETS MADE IN TAP WATER AND IN DEIONIZED WATER[a]

Sample No. 3101-	Uranium 8/5	Uranium ng/g	Lithium, ng/g	Description
393	127	1.35	41.1	Handsheets made from Hercules pulp, PS-57, in 6 liters of tap water (Appleton city water, pH 9.5)
394	121	0.71	5.55	Handsheets, same as 393 except that each handsheet was rinsed on the mold with 2 x 200 ml of deionized water
396	119	0.86	4.28	Handsheets, same as 393 except that each handsheet was rinsed on the mold with deionized water at pH 9 (ammonium carbonate)
397	127	1.02	44.4	Handsheets made from purified pulp No. 341 (see Report Fourteen, p. 28), in 6 liters of tap water (Appleton city water, pH 9.5)
403	127	0.27	4.98	Handsheets, same as 397 except that each handsheet was rinsed on the mold with 2 x 200 ml of deionized water
404	118	0.47	3.67	Handsheets, same as 397 except that each handsheet was rinsed on the mold with deionized water at pH 9 (ammonium carbonate)
407	133	0.11	3.07	Handsheets, same as 397 except that deionized water at pH 9 only was used in making the handsheets
405	117	0.14	0.58	Hercules pulp, PS-57, purified by leaching with hydrofluoric acid and ammonium carbonate, one cycle; used for couching handsheets in Sample 407
390	138	0.149 ng/ml	11.1 ng/ml	Appleton city water, pH 9.6
391	116	1.29	2.36	Hercules pulp, PS-57; control [same as 3101-001, Report Seven, p. 7]
341 C	119	0.041	0.26	Purified Hercules pulp (PS-57) [see Project 3101, Report Fourteen, p. 28]

[a]Analytical data were received by letter dated February 5, 1975, from Jack Phelps to E. E. Dickey.

consistency, retained >40 ng Li/g of which at least 30 ng was due to process water dried on the sheet. This premise was reinforced by Samples 394 and 396 which were made in tap water and rinsed on the sheet mold with deionized water and with water adjusted to pH 9 with ammonium carbonate, respectively. The removal of 90% of the lithium by washing with purified water was consistent with the results of the limited leaching experiment, Samples 385A to Y, Table III.

Based on the results of these handsheet studies, a rinsing of the sheet on the wire of the fourdrinier machine with purified (deionized or distilled) water might reduce significantly the level of lithium in commercially-made IPC-1478 paper. The use of purified pulp made into handsheets in deionized water (407) showed some advantage over the handsheets made from purified pulp in tap water and rinsed with purified water (403) or ammonium carbonate (404).

RECOVERY OF FINES FROM SERIES N PAPER

Although the role of fines in the retention of lithium by IPC-1478 paper had been explored briefly, both at the Institute (3) and at McClellan AFB, MCL-C (see Appendix II, p. 45), further work seemed necessary. The chief question was: Is lithium bound in the fines in an inaccessible or water-insoluble form as compared with that in the pulp?

In the Series 412 to 414, 421, and 422, shown in Table V, fines were loosened from Series N paper (minus the scrim) by stirring in a British Disintegrator*. The fines were drained from the pulp by pouring the slurry into a

*For a description see TAPPI Standard Method T 205 os-71 (TAPPI, 1 Dunwoody Park, Atlanta, GA 30341).

TABLE V

COLLECTION AND ANALYSIS OF FINES FROM PS-57 PULP AND IPC-1478 PAPER, SERIES N[a]

Sample No. 3101-		Weight Analyzed, g	Uranium 8/5	Uranium ng/g	Lithium, ng/g	Remarks
Hercules Pulp, PS-57, was dispersed in deionized water, the slurry was poured into a plastic funnel (no filter paper was used), the "white water" was collected and then filtered on a circle of the pulp in the dry-lapped form. Each circle carried the fines from 60 g of pulp.[b]						
298	A	5.460			2.66	Circles No. 2, 3 bearing fines
	B	5.243			2.75	(0.20 g estimated) from pulp
		Av. 5.351			Av. 2.71	Samples 299 and 300
Concentration of lithium in fines (298), only					38	By approximation[c]
299	A				1.25	Pulp pads from which fines 298
	B				1.32	were recovered
					Av. 1.29	
303	A	5.111			2.80	Circles No. 5, 6 bearing fines
	B	5.559			3.02	(0.20 g estimated) from pulp
		Av. 5.335			Av. 2.91	Samples 304, 305
Concentration of lithium in fines (303), only					43	By approximation[c]
304	A				1.28	Pulp pads from which fines 304
	B				1.32	were recovered
					Av. 1.30	
IPC-1478 Paper, Series N, scrim removed, dispersed in deionized water, slurry poured on a plastic screen. The "white water" filtered on tared circle of purified Hercules PS-57 pulp, No. 341. Five lots processed; water was recycled.						
413		5.04	132	0.88	7.33	Fines 0.183 g, from 414 on 4.86 g purified pulp
Concentration of lithium in fines (413), only					125	Calculated[d]
414		13.8	130	1.05	2.79	54 g Pulp from Series N paper after fines (413) removed
421		5.22	132	1.08	14.4	Fines, 0.175 g, from 422 on 5.04 g purified pulp
Concentration of lithium in fines (421), only					107	Calculated[d]
422		14.0	124	0.66	10.8	55 g Pulp from Series N paper after fines (421) removed
412		200 ml	133	0.0054	2.10 ng/ml	Filtrate, 3.8 liters, at end of fifth lot of fines (421)
IPC-1478 Paper, Series N. The procedure described above for Sample No. 412-422 was repeated except that the "white water" was not recycled.						
435		5.21	130	0.584	5.47	Fines from No. 436, 0.218 g on pulp circle (341)
Concentration of lithium in fines (435), only					39	
436		9.02	128	0.844	3.83	Fiber from Series N paper
437		200 ml	--	--	0.204 ng/ml	Filtrate from 435

[a] These data in part were reported in a letter dated April 24, 1975, from Jack Phelps, McClellan AFB, to E. E. Dickey.
[b] For details of this procedure see Project 3101, Report Fourteen, p. 47.
[c] The lithium contents were approximated by assuming that the weight of fines was 0.200 g on each circle, and that the lithium not held in the fines was the same as that in the pulp samples, 299 and 304, respectively.
[d] The lithium contents were calculated on the assumption that the lithium not held in the fines was the same as that in the pulp samples, 414 and 422, respectively.

plastic funnel fitted with a plastic screen. The "white water" was then filtered through a tared circle of purified PS-57 pulp, and the amount of fines was determined gravimetrically.

To determine the amount of lithium associated with the fines, only (and not with the pulp circle), an assumption was made that the lithium on the pulp circle fibers was the same as found on the pulp pad from which the fines had been dislodged. Based on this assumption, the amount of lithium in the fines was estimated. As an example, the amount of lithium in the fines from Sample 298 was calculated from the data in Table V:

$$\frac{\left[\begin{matrix}\text{av. wt. (g)} \\ \text{of Circle 298}\end{matrix}\right] \times \left[\left(\begin{matrix}\text{av. Li content} \\ \text{of 298}\end{matrix}\right) - \left(\begin{matrix}\text{av. Li content} \\ \text{of 299}\end{matrix}\right)\right]}{[\text{wt (g) estimated of fines in 298}]} = \text{Li content of fines, only,}$$

or $$\frac{5.351\ (2.71 - 1.29)}{0.20} = 38 \text{ ng Li/g of fines, only.}$$

The corresponding values for 303, 413, and 421 showed that, compared with pulp from the same system, a disproportionately large concentration of lithium was present in the fines. However, the amount of lithium carried in the fines would have added <20% of the lithium retained in the residual pulp. Even so, a closed white water system for the papermaking process to produce low-lithium IPC-1478 paper may be improved if a suitable step were placed in series to remove fines.

In an effort to gain more knowledge about the possible role of fines in lithium retention, fines were collected from Series N paper by the general procedure, as reported previously, except that the process water was not recycled.

The experiment was designed to expose the fines to the lowest possible concentrations of lithium. As shown in Sample 436, Table V, the fines retained 10 times the concentration of lithium held by the residual fiber (436).

ATTEMPTED STUDY OF LITHIUM ACCESSIBILITY WITH LITHIUM-6

Series N paper (scrim removed) was dispersed in water, the dislodged fines were collected on circles of purified pulp (341), and both the fiber and the fines were exposed to an aqueous solution of lithium-6. The analyses are listed in Table VI. The labeled material was washed with water (464 and 465), and leached with hydrofluoric acid and ammonium carbonate (468 and 469). Concentrations of lithium in the fines, only, were estimated by the scheme described previously on p. 16-18.

The results were generally consistent with results obtained earlier (4). Thus, the fiber obtained after dislodging the fines was washed with water, only, and retained 2.7-3.0 ng Li/g (457, 461, 465). When the fiber was labeled with lithium-6 (465), and then washed with water, a small amount of the lithium-6 label remained in the sample as indicated by the 6/7 ratio, 0.1026, compared with natural lithium at 0.08. The fines retained a larger proportion of the lithium (464) with a 6/7 ratio of 13.08. However, when both the fiber and the fines were leached with hydrofluoric acid and ammonium carbonate, the levels of lithium were similar to those generally expected in such purifications (468 and 469). The 6/7 ratio was nearly that of natural lithium for the sample of fiber (469), whereas that of the fines (468) was 0.2295, indicating a possible exchange of some of the persistent lithium with the label.

TABLE VI

ACCESSIBILITY TO LITHIUM-6 OF FIBERS AND FINES OF SERIES N PAPER

Sample No. 3101-	Weight Analyzed, g	Uranium 8/5 ng/g		Lithium 6/7 ng/g		Remarks	
IPC-1478 Paper, Series N (scrim removed) was dispersed in deionized water, and the fines were collected on circles of purified pulp (341).							
457	10.30				2.79	Paper fiber from No. 458	In water, only
458	5.218				4.70	Pulp circle with fines (0.127 g)	
Concentration of lithium in fines, only					78.5		
461	9.014				3.00	Paper fiber from No. 462; water only	
462	5.529				2.22	Pulp circle with fines (0.134 g); leached with hydrofluoric acid and ammonium carbonate	
Concentration of lithium in fines, only					(28.6)[b]		
465	12.87	132	1.30	0.1026	2.74	Paper fiber from No. 464	Exposed to lithium-6 and washed with water, only
464	5.32	123	0.352	13.08	41.4	Pulp circle with fines (0.145 g)	
Concentration of lithium in fines, only					1420		
466[a]	200 ml	135	0.00094	27.64	0.698	Filtrate from No. 465	
469	12.4	134	0.112	0.08714	1.66	Paper fiber from No. 468	Exposed to lithium-6 and leached with hydrofluoric acid and ammonium carbonate
468	4.86	135	0.119	0.2295	1.81	Pulp circle with fines (0.117 g)	
Concentration of lithium in fines, only					6.23		

[a] Uranium and lithium analyses listed in ng/ml.

[b] The calculation was based on the assumption that the fiber in the pulp circle retained lithium in a concentration of 1.5 ng Li/g.

The results support the earlier experiences (4) that most if not all the lithium may be exchangeable. However, the reason for the higher concentrations of lithium in fines as compared with the corresponding fiber is unknown. Generally, the fines carried only a small fraction of the total load of lithium in the IPC-1478 papers and may be of minor significance in the making of low-lithium papers.

POSSIBLE EFFECT OF TEMPERATURE ON THE PURIFICATION PROCESS

Samples of both pulp and Series N paper were leached with reagents and water heated to 80°C for comparison with similar samples leached at ambient temperatures (20-30°C). The uranium and lithium contents are shown in Table VII. No significant advantage was evident for the use of heated reagents and wash water in the purification process.

TABLE VII

URANIUM AND LITHIUM CONTENTS OF PS-57 PULP AND SERIES N PAPER PROCESSED AT 80°C

Sample No. 3101-	Uranium 8/5	Uranium ng/g	Lithium, ng/g	Remarks
480	118	1.34	0.938	PS-57 pulp
482	129	0.856	3.13	Series N paper

ANALYSES BY EMISSION SPECTROGRAPHY

The mineral contents of selected samples of IPC-1478 paper and Hercules pulp, PS-57, from which the paper was manufactured, were determined by emission spectrography. The results, summarized in Table VIII, show that 85-91% of the minerals (ash components) was removed in the purification process. The increased amounts of sodium and calcium of the paper (No. 294) compared with the pulp (No. 001) are believed to be due in part to the chemicals used in treating the water in the mill at Watertown, NY. The increase in iron and copper may be due to

TABLE VIII

ANALYSES OF IPC-1478 PAPER, SERIES N, AND HERCULES PULP, PS-57, BY EMISSION SPECTROGRAPHY

The samples were ashed at 550° in a muffle furnace

Sample	IPC-1478 Paper, Series N, Roll No. 22			Hercules Pulp, PS-57			
No. 3101-	294, control	279, 281 purified	Residue After Purification, %	001, control	341, purified	Residue After Purification, %	Chemically Treated[c]
Amount analyzed, g	4.68[a]	6.60[a]		13.70	12.15		4.15
Yield of ash, %	0.11	0.017	15	0.056	0.0051	9	0.13
Elements by Emission Spectrography, ppm							
Aluminum	14	2.2	16	9.8	0.54	5.5	3.4
Barium	0.58	0.15	26	0.23	0.047	20	0.20
Boron	0.57	0.074	13	0.056	0.13	230	1.4
Calcium	128	32	25	99	8.6	8.7	18
Chromium	0.16	0.096	60	0.11	0.029	26	0.28
Copper	4.3	0.42	9.8	1.4	0.40	29	7.5
Iron	18	6.8	38	5.2	1.5	29	9.9
Lead	0.23	0.12	52	0.16	0.058	36	0.68
Magnesium	11	3.6	33	11	1.8	16	520
Manganese	0.94	0.040	4	0.15	0.016	11	0.35
Phosphorus	8.8	3.5	40	4.0	1.6	40	--
Silicon	22	7.2	33	18	2.4	13	50
Silver	0.0089	0.0089	100	0.0052	0.0024	46	--
Sodium	250	16	6.4	86	14	16	32
Titanium	0.64	0.41	64	0.32	0.16	50	0.78
Lithium, ng/g[b]	23.4	1.03	4	2.37	0.37	16	--
Uranium, ng/g[b]	0.64	0.06	9	1.26	0.05	4	--

[a] These samples were ashed without removing the scrim. At McClellan AFB the scrim is removed before samples of paper are analyzed.

[b] Data for lithium and uranium from Project 3101, Report Seven, p. 7 and Report Fourteen, p. 28, 31 (No. 280).

[c] The Hercules pulp was treated with 1% magnesium carbonate and 5% sodium hydroxide (based on the weight of the pulp) at 120° for one hour, washed with deionized water and dried.

the equipment and plumbing. The increases in aluminum and silicon were not marked, and most other elements were not changed significantly.

The purified samples (No. 279, 281, and 341, respectively) retained proportionately larger amounts of the main ash components than of the minor elements. Thus, sodium, calcium, iron, silicon, and phosphorus were especially persistent, and readily account for the bulk of the ash in the purified pulp and paper.

The uranium and lithium data for these samples were reported previously (1,2), but are included in Table VIII for comparison. It may be noteworthy that the efficiency of the removal of lithium (96%) was approximately the same as that of sodium (94%) for the IPC-1478 paper. The efficiency for the removal of lithium (84%) from the pulp was lower than for the paper, but the total amount was approximately one-tenth of that of the paper. The efficiency of sodium removal (84%) was similar to that of lithium.

The removal of uranium was accomplished at efficiencies of 91-96%, similar to the removal of manganese. This observation, together with the observed similarity between lithium and sodium, may be considered in the design of experiments which might afford the monitoring of some of the results of purification attempts. Thus, sodium and manganese may be considered as indicators for lithium and uranium, respectively.

It was observed at the McClellan Laboratory that the ash formed from the Series N paper was brown colored, whereas the ash from the pulp samples was white. The spectrographic analyses failed to reveal components which might account for the dark color and the cause remains unknown.

In the last column in Table VIII are listed the components in a sample of Hercules PS-57 pulp which had been heated for one hour at 120° with magnesium carbonate and sodium hydroxide, washed with water and dried*. The results indicated that even without an acid treatment, the levels of aluminum, calcium, and sodium were reduced relative to the starting material (001). Magnesium, which was probably absorbed on the pulp fibers, was the most persistent element under the conditions of the experiment. Also, the increased levels of silicon, boron, iron, and copper may be due to contact with glass and stainless steel apparatus. In general, the behavior of the pulp components in this experiment was consistent with predictable behavior.

PILOT PLANT STUDIES, KNOWLTON BROTHERS CO., AUGUST, 1974

IPC-1478 paper, Series N, was made by Knowlton Brothers Co., Watertown, NY, in October, 1973. Subsequently, the lithium content was found to be approximately 20 ng Li/g, a level too high for use in most field applications. In order to try to determine the reason(s) for the lithium in the Series N paper, pilot-plant experiments were designed by personnel of the McClellan AFB, MCL-C in cooperation with Knowlton Brothers. The tests were conducted August 11-14, 1974 (see Appendix II).

The results of these tests support the premise that the high lithium level of the Series N paper was due to the unexpectedly high levels of lithium in the process water. Details of the experiments and of the results were recorded at McClellan AFB, MCL-C, under the supervision of Jack Phelps, Chief, Mass Spec Chemistry Unit, who may be consulted for further information.

*PS-57 pulp was used as "pure" cellulose on a project (IPC) which supplied the data in Table VIII, Column 8.

AIR-FLOW AND RETENTION EFFICIENCY TESTS OF PURIFIED IPC-1478 PAPER

Selected samples of purified IPC-1478 paper were sent to Knowlton Brothers where, under the supervision of Dr. Grant Rauscher, air-flow and particle retention efficiency tests were obtained. The results as supplied by Dr. Rauscher are placed in Appendix III of this report; a copy was sent to Jack Phelps at MCL-C, McClellan AFB.

The results indicated no significant changes were introduced into the air-filtering characteristics due to the purification procedures.

CONCLUSIONS

TWO POSSIBLE PROCESSES FOR THE PREPARATION OF LOW-URANIUM, LOW-LITHIUM FILTER PAPER OF THE IPC-1478 TYPE

Based on the results of research and testing over the three-year period of the contract, July 1, 1972 to June 30, 1975, two processes were developed by which an IPC-1478 type of paper may be produced with low levels of uranium and lithium. Uranium was reduced to levels of <0.1 ng U/g in both the IPC-1478 paper and the pulp from which it was made. However, the 8/5 ratio was changed from 118 in the pulp to the common natural level of 130-140 in most of the purified products. These properties were well within the specifications set by the U.S. Air Force at <0.3 ng U/g with an 8/5 ratio of natural uranium, 137.

Lithium proved to be much more persistent than uranium, with commercially-made IPC-1478 paper somewhat more tenacious in holding the lithium than was the Hercules cotton linters pulp, PS-57. Thus, handsheets made from purified pulp (0.15-0.3 ng Li/g) under the best conditions available, retained 0.7-0.8 ng Li/g, and purified IPC-1478 paper (commercial grade) retained 1-2 ng Li/g, thereby exceeding the lithium specification set by the U.S. Air Force at <0.4 ng Li/g in finished paper. At present, the lowest practical level for lithium would appear to be approximately 1 ng Li/g.

PROCESS NO. 1. PURIFICATION OF COMMERCIALLY-MADE IPC-1478 PAPER

a. Place a stack of 100-200 squares (any size required) of the IPC-1478 paper in a suitable tray.

b. Leach the paper by percolation under gravity with 1 liter of each of the following in sequence for each 250 g of paper:

0.1M hydrofluoric acid

deionized water

0.1M ammonium carbonate

deionized water

0.1M hydrofluoric acid

c. Wash the stack with 7 liters of deionized water.

d. Press the stack to a consistency of 20-25% at reduced pressure under a rubber sheet.

e. Dry the paper in filtered air.

f. Repeat the purification cycle, a-e.

g. Apply 30% Kronisol by weight of the airdry sheet.

PROCESS NO. 2. IPC-1478 PAPER MADE BY HAND FROM PURIFIED PULP

a. Purify dry-lapped pulp by the process described in Part 1, a-c, inclusive.

b. Purify a roll of scrim (wound on an inert core) by the process described in Part 1, a-c, inclusive.

c. Without drying the pulp, disperse it in deionized water adjusted to pH 9 with ammonium carbonate and ammonium hydroxide.

d. Form sheets on scrim in a sheet mold. For 250 g of handmade paper, 500-600 liters of water would be required. Rinse each sheet twice on the wire with small portions of water, couch with purified pulp sheets, and dry in filtered air.

e. Apply 30% Kronisol by weight of the airdry sheet.

GENERAL REQUIREMENTS

Both processes can be located in a single installation with the following capabilities:

a. All new, stainless steel and plastic equipment.

b. Supply of deionized water with <0.01 ng U and Li/ml.

c. All equipment and operations in a room with filtered air at a slight, positive pressure.

d. A dryer capable of drying 100-200 sheets (in sets of three sheets) per 24-hour day in a stream of filtered air at room temperature or at elevated temperature.

e. Roll applicator for Kronisol.

The continued use of the facilities would scour the levels of all impurities to the lowest practical limits. Based on the experiences on Project 3101-1, uranium would be consistent at approximately 0.1 ng/g and lithium would be expected to level off at approximately 1.0 ng/g of finished paper.

No attempt has been made, herewith, to provide specifications on the size and capacity of the installation. Such specifications must be based on the production rate of the paper required in accord with the needs of the U.S. Air Force.

EXPERIMENTAL

IPC-1478 FILTER PAPER, SERIES N

All commercially-made IPC-1478 paper used on Project 3101-1 was designated Series N, and was made in October, 1973 by Knowlton Brothers Co., Watertown, NY. None of the paper had been treated with Kronisol. Each of thirteen rolls was cut into approximately 120 sheets 19 inches square. The stack of squares from each roll was treated as a unit and weighed 3.84 kg (8.47 lb).

KRONISOL

One five-gallon lot of Kronisol (dibutoxyethyl phthalate) was furnished to Project 3101 by the Knowlton Brothers Co. in 1973. The compound was manufactured by C. P. Hall Co., Chicago, IL. The uranium and lithium contents are listed in Table II, Sample 392.

SAMPLE NUMBERING SYSTEM

As described in Report Seven, p. 25, samples were assigned unique numbers by the Institute in the Series 3101-001 to 3101-999 as suggested by Captain Frank Grosso. These numbers are composed of a four-digit number 3101, the same as the IPC project number, followed by a unique three-digit number. Throughout the reports, the four-digit prefix was omitted for the sake of simplicity; the last number assigned in the series was 488.

ANALYSIS OF SAMPLES

The analysis of all samples for uranium and lithium was done under the direction of Mr. Jack Phelps, McClellan AFB, MCL-C. All analytical data are uncorrected for blanks.

365. PURIFICATION OF SERIES N PAPER, ROLL 22

An amount of 35 sheets, 20 inches square, was cut by hand from Roll No. 22 of the IPC-1478 paper, Series N. Several sheets of the Hercules PS-57, dry-lapped pulp were stacked with the paper, and the stack was leached in succession by percolation with the following solutions.

8 liters 0.1M hydrofluoric acid
10 liters deionized water
8 liters 0.1M ammonium carbonate
10 liters deionized water
8 liters 0.1M hydrofluoric acid
20 liters deionized water.

The stack was pressed under a rubber sheet at reduced pressure (approximately 10 inches of mercury produced by a water aspirator pump), and dried in a stream of filtered air at room temperature.

Kronisol, 362 g (30% based on 1206 g of the dry, purified paper), was poured as evenly as possible onto the stack of purified paper. After standing for two hours, the bottom sheets were interleaved with the top sheets and the stack was allowed to stand for 48 hours. The sheets were then weighed individually and restacked with the sheets containing the least Kronisol placed in contact with

those containing the largest amount of Kronisol. In this order the stack was assigned the number 365 and was shipped to McClellan AFB. Representative analytical data are listed in Table II.

PURIFICATION OF SERIES N PAPER, 19-INCH SQUARES*

LIST OF SAMPLE NUMBERS OF PURIFIED SERIES N PAPER

Numerals in () identify the lot number, Table II, p. 9

370(3)	372(4)	423(6)	374(5)	425(5)	366(1)	427(7)	368(2)	389(7)
410(5)	472(9)	473(9)	474(10)	475(10)	376(2)	477(11)	378(3)	409(4)
430(8)		483(11)	484(11)	485(12)	386(6)		398(8)	429(8)
470(9)					406(4)		428(7)	
					426(6)			
					476(10)		488(12)	
					486(12)			

Stacks of 19-inch squares, 100-120 sheets from each roll, were leached by percolation in succession with 15 liters of each of the following solutions in two cycles.

a. 0.1M hydrofluoric acid

b. deionized water

c. 0.1M ammonium carbonate

d. deionized water

e. 0.1M hydrofluoric acid

f. deionized water (55 or 95 liters)

*See Process No. 1, p. 27.

The stack was pressed under a rubber sheet by a partial vacuum (water aspirator pump) to a consistency of 20-25% and the sheets were dried in a stream of filtered air at room temperature. The leaching steps required approximately 5 hours, except when the schedule was extended to 3 or 8 days. In the latter cases, a total of 95 liters of deionized water was used to wash the paper in portions of 25, 30, 20, and 20 liters, one or two portions per day on successive days, in place of one 55-liter portion.

Kronisol was applied from a small paint sprayer (2) or from a perforated polyethylene (PE) bottle at a rate of 30% based on the weight of the untreated, airdry paper.

The PE bottle (500-ml, screw-capped) was perforated with a needle (0.7 mm in diameter) in a narrow band, 5 mm in width, top-to-bottom, on the cylindrical part of the wall, only. Approximately 150 holes formed the perforated zone. The bottle was filled half or two-thirds full of Kronisol, the perforated zone was placed in contact with the paper and, using quick, even strokes (with a light pressure by hand on the bottle) Kronisol was applied as evenly as possible to each sheet. Each sheet was weighed (but not recorded) to monitor the addition of Kronisol. Analytical samples and the finished paper were assigned the numbers shown in Table II and in the list on p. 32.

380-384. PURIFICATION OF IPC-1478 PAPER BY LEACHING INTACT ROLLS

Rolls of IPC-1478 paper, Series N, were provided by the laboratory at McClellan AFB. Each roll (10 inches by 80 feet) was wound on a length of PVC sewer pipe (3-1/2 inches OD) to minimize contamination. With the roll in a

horizontal position in the leaching tray, it was leached by percolation with 4 liters each of the following reagents in succession:

a. 0.1M hydrofluoric acid

b. deionized water

c. 0.1M ammonium carbonate

d. deionized water

e. 0.1M hydrofluoric acid

f. deionized water.

The roll was pressed under a rubber sheet, was draped in an accordion fold on a drying rack, and was dried for 24 hr in a stream of filtered air at room temperature. Samples for analysis were removed near each end of the dry strip of paper, the remainder was rewound, and the leaching cycle was repeated. The data are summarized in Table III.

385A to Y. LIMITED LEACHING OF IPC-1478 PAPER

A stack of 100, 17-centimeter squares of Series N paper (Roll 22) was placed on the leaching platform and deionized water in small portions was poured onto the stack. By percolation and capillary movement, just sufficient water (1300 ml) was used to wet the stack. Without pressing, the stack was separated into sets of four sheets and the sets (A to Y) were dried in a stream of filtered air at room temperature. The analysis of selected samples is listed in Table III.

399-402. ATTEMPTED PURIFICATION OF IPC-1478 PAPER BY CAPILLARY MIGRATION OF LEACHING AGENTS

Strips (24 cm by 110 cm) of IPC-1478 paper, Series N, Roll 22 were placed in a glass tank as commonly used for paper chromatography. One trough

was all-polyethylene and the other all-glass. After approximately 1 liter of deionized water passed through the sheet by capillarity, the sheet was dried in the air (unfiltered) at room temperature. Additional strips of paper were leached with two one-liter portions of water with an intermediate drying step, and one set of strips was leached with 1 liter each in succession of 0.1M ammonium carbonate and water. Analytical data are recorded in Table III.

390 to 394, 396, 397, 403, 404, 407. HANDSHEETS MADE IN TAP WATER AND IN DEIONIZED WATER

Hercules cotton linter pulp, PS-57, was dispersed in tap water (Appleton city water, pH 9.6) in a British Disintegrator* at the rate of 40 g airdry pulp in 2 liters of water. The disintegrator was operated for 5 min (600 counts, 15,000 revolutions of the stirrer) for each 40-g batch of pulp. Four 40-gram batches were combined and diluted in a stainless steel tank to a total volume of 32 liters, 0.5% consistency. For each handsheet, 1200 ml of the stock containing 6 g of airdry pulp, was diluted to 6 liters in a sheet mold and formed into a handsheet 8 inches square. Each sheet was couched with squares of pulp, pressed, and dried in filtered air at room temperature.

Sets of handsheets were made from tap water, washed on the sheet mold twice with 200-ml portions of deionized water and with water at pH 9 (ammonium carbonate).

Other sets of handsheets were made from purified pulp (341) in tap water and in deionized water.

*See footnote p. 16.

The analytical data are listed in Table IV.

413-422, 435-437. RECOVERY OF FINES FROM SERIES N PAPER

The scrim was removed from 250 g of Series N paper which was then dispersed in 25-g batches in a British Disintegrator*. Each 25-gram batch was stirred for 5 min (600 counts, 15,000 revolutions of the stirrer) in deionized water adjusted to pH 9 with ammonium carbonate, the slurry was poured into a plastic funnel with a plastic screen septum, the pulp pad was redispersed in an additional 2 liters of water at pH 9, and the slurry was poured again into the plastic funnel. The pulp pad was pressed under a rubber sheet at reduced pressure (water aspirator pump) and dried. A tared circle of purified pulp (341) was placed on the funnel and the 4 liters of white water combined was filtered through the circle of pulp to collect the fines. The circle was not washed or pressed before drying. The process was repeated except that the same tared circle of pulp was used for the collection of the fines thereby combining the fines from 50 g (excluding scrim) of the original Series N paper.

Four additional 50-g lots of the Series N paper were processed by the above procedure using recycled filtrates from the fines filtration step. Analytical data are summarized in Table V.

Another set of fines (435-437) was collected from Series N paper according to the general procedure described above except that the filtered white water was not recycled. The analytical data are listed in Table V.

*See footnote p. 16.

438-444. LEACHING OF IPC-1478 PAPER BY PERCOLATION WITH CAPILLARY FEED

Series N paper, 25 sheets (15.2-cm squares), was stacked between two sets, of six squares each, of dry-lapped pulp (Hercules PS-57). Additional sheets of pulp (15 by 50 cm) one on the top and one on the bottom of the stack were added as wicks to transport the reagents into the stack (top wick) and to remove the process fluids from the stack (bottom wick). The stack was placed on a Lucite platform such that the top wick was held in a plastic (PE) vessel fed by an automatic siphon (inverted bottle). The bottom wick delivered the wastes to the drain. Over a period of twelve days the stack of paper and pulp was leached successively with one liter each of 0.1M hydrofluoric acid, water, and 0.1M ammonium carbonate followed by 14 liters of water. The stack was pressed under a rubber sheet at reduced pressure and dried in a stream of filtered air at room temperature. The analytical data are listed in Table III.

445-454. PURIFICATION OF HERCULES PULP, PS-57, AND THE COLLECTION OF FINES

A. Purification of the Pulp

Hercules pulp, PS-57, 250 g, was dispersed in 25-gram batches in 2 liters of deionized water by stirring in a British Disintegrator for 600 counts (15,000 revolutions of the stirrer). The pulp was carefully pressed to the bottom of the stirring vessel with a Lucite plunger (many 5-mm holes) and the supernatant was decanted into a 30-liter plastic wastebasket. The pulp was diluted to 2 liters with water, stirred for 30 counts in the disintegrator and the slurry poured into a plastic funnel. The funnel was made from a 4-liter, cylindrical bottle with the bottom cut away. The septum was made by trimming the bottom to fit inside the bottle, and by drilling uniformly 80-100

1-mm holes. The excess liquid was removed under partial vacuum and the pulp pad was pressed lightly with a plastic (PE) stirring rod. Each succeeding 25-gram batch was added in the same manner until a thick mat containing 250 g of airdry pulp had accumulated.

The pad was leached by percolation under gravity with 3 liters each of 0.1M hydrofluoric acid, water, 0.1M ammonium carbonate, water, 0.1M hydrofluoric acid and water over a period of two days. Over the next eight days fifteen, 3-liter portions of deionized water were percolated through the pad. The total time of treatment required ten days and the total amount of wash water was 48 liters. The purified pulp was pressed under a rubber sheet at reduced pressure, separated into eight pads as originally formed and dried. The analytical data are summarized in Table III.

B. Collection of Fines

The combined white water from Part A (see above) was filtered through tared circles of purified pulp. The yield and analysis of the fines are summarized in Table III.

455-469. SERIES N PAPER. ACCESSIBILITY IN THE PURIFICATION PROCEDURE OF FIBERS AND FINES TO LITHIUM-6

A. Preparation of Fiber Pads and Fines

Series N paper (scrim removed), 25 g, was dispersed in 2 liters of water in a British Disintegrator* at 600 counts (15,000 revolutions of the stirrer). The pulp was concentrated at the bottom of the stirring vessel with

*See footnote p. 16.

a perforated plunger and the supernatant was filtered through a plastic funnel equipped with a plastic screen (100 mesh) septum to produce fiber-free white water. The pulp mass was dispersed again in 2 liters of water and the dispersion step was repeated. After the supernatant had been filtered and combined with the first volume of white water, the pulp as transferred to the funnel in 500 ml of slurry, formed into a pad, pressed under a rubber sheet, and dried. The entire procedure was repeated with another 25-g sample.

The combined white water (<8 liters from 50 g fiber) was filtered through a tared circle of purified pulp and the filtrate was discarded. The analytical data are summarized in Table VI.

B. 459-462. Fines Leached with Hydrofluoric Acid and Ammonium Carbonate

The procedure described in Part A (see above) was repeated except that the wet circle bearing the fines was leached in sequence with 200 ml each of 0.1M hydrofluoric acid, water, 0.1M ammonium carbonate, water, 0.1M hydrofluoric acid, washed with 500 ml of water, and dried (without pressing). The entire experiment was repeated. The analytical data are summarized in Table VI.

C. 463-469. Fines and Paper Fiber Labeled with Lithium-6

The procedure described in Part A (see above) was repeated except that the wet circle bearing the fines was placed in a plastic beaker containing 200 ml of water to which 5000 ng of lithium-6 (as $^{6}Li_2CO_3$, >99% ^{6}Li) had been added. The fiber pad, from which the fines had been collected, was dispersed in 2 liters of water to which 5000 ng ^{6}Li had been added. The fines were exposed to the lithium-6 overnight, and were washed with water and dried.

The above labeling procedure was repeated except that the circles bearing the fines were purified as in Part B. The combined paper fiber pads were leached with 1 liter, each, of the reagents and water, washed with 4 liters of water, and dried. The analytical data are summarized in Table VI.

479-482. POSSIBLE EFFECT OF TEMPERATURE ON THE REMOVAL OF LITHIUM IN THE PURIFICATION OF IPC-1478 PAPER AND HERCULES PULP, PS-57

A. Pulp Samples

Hercules pulp, PS-57, 50 g, was dispersed in 2 liters of water by stirring in the British Disintegrator for 300 counts (7500 revolutions of the stirrer). The slurry was poured into the plastic funnel (PE bottle, see p. 37), and excess water was removed under reduced pressure. The step was repeated twice and the pulp was combined in the funnel for a total of 150 g. The thick mat of pulp was then washed by percolation with 10 liters of deionized water at room temperature. The mat of pulp was pressed under a rubber dam, separated into 50-g pads and dried.

The above experiment was repeated except that the final washing was done with 10 liters of water at 80°C. The pulp samples were not submitted for analysis.

B. Series N Paper

Three 50-g batches of Series N paper (scrim removed) were processed according to the procedure in Part A (see above) except that the pulp mat (150 g) was leached by percolation with 2 liters each of the reagents and water as described for Samples 445-454, p. 37. The experiment was repeated except that all reagents and wash water were heated to 80°C. The analytical results are listed in Table VII.

ACKNOWLEDGMENTS

In behalf of the Institute personnel, especially the undersigned, appreciation and gratitude are expressed to the personnel at the McClellan AFB, MCL-C. Without the analytical data supplied to the Institute from the mass spectrographic laboratory, Project 3101 would have been impossible. In particular we appreciate the courteous help of Captain Frank Grosso and Jack Phelps.

LITERATURE CITED

1. Project 3101, Report Seven.

2. Project 3101, Report Fourteen

3. Reference 2, p. 33.

4. Reference 1, p. 14-17.

THE INSTITUTE OF PAPER CHEMISTRY

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APPENDIX I

STATEMENT OF WORK TO BE DONE
(AFTAC Project Authorization No. T/5208/L/ETR)

1.0 Objective: The objective of this contract is to use the projection technology developed in Project T/4208 to produce paper having a uniform contaminant content at the specified low levels. This paper is then to be sampled, tested, and evaluated to insure its suitability as a substitute for IPC-1478 paper.

2.0 Tasks:

2.1 The contractor will decide upon which manufacturing procedure of those investigated under Project T/4208 will be used to produce a paper for sampling, testing, and evaluation with the following properties:

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

2.1.2 Uranium content not to exceed 0.3 ng U/gm of paper with a U238/U235 atom ratio approximately equal to 137.8 (natural value). Furthermore, the manufacturing procedure should be capable of the consistent production of low-lithium, low-uranium filter paper with filtering properties similar to IPC-1478 filter paper.

2.2 Paper samples, cotton linters, and process reagents will be submitted to the McClellan Central Laboratory (MCL) for uranium and lithium analyses. MCL will also arrange for the testing and evaluation of the filtering characteristics of the new paper.

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

2.2.2 To avoid contamination by handling, all filter paper samples should be handled with non-talc rubber or plastic gloves and shipped in special plastic bags supplied by MCL.

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

2.2.4 All liquid solutions should be shipped in clean polyethylene bottles and contain approximately one liter of solution. Bottles should be labeled.

2.3 Papers produced for sampling, testing, and evaluation will be cut into 4.25-inch diameter circles and separately marked. These samples will be stored and shipped in plastic bags supplied by MCL.

2.3.1 To guarantee that this paper has similar filtering properties to IPC-1478, all manufactured paper will be impregnated with Kronisol (dibutoxyethyl phthalate), 30% by weight and backed with a cotton bleached guaze scrim, 18 x 14 threads/inch.

2.3.2 The contractor will procure both the Kronisol and scrim. These will be evaluated and approved by MCL prior to use in this project.

2.4 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 will be submitted within three months after completion of work. The report should detail the final manufacturing procedures, the results of the MCL sampling, testing, and evaluation, and any other experiments performed during the project. Negative results will be reported.

APPENDIX II

PROJECT 3101-1 MEMORANDUM.
CONFERENCE AT KNOWLTON BROTHERS CO., WATERTOWN, N.Y.,
AUGUST 11-14, 1974. PILOT-PLANT TESTS

Summary

The first-hand observation of the pilot plant tests, and the discussions with personnel from Knowlton Brothers and from McClellan Air Force Base were very helpful in our understanding of the nature and origin of uranium and lithium contents of IPC-1478 paper.

1. The uranium contents of IPC-1478 papers resulting from the papermaking process are due mainly to the levels of uranium in the pulp and, to some degree, in the process water.

2. The relatively high lithium content of Series N paper appears to have come mainly from the lithium carried (on the sheet) by the process water into the drying step.

3. The relatively high lithium content of the process water (five times higher than the Watertown city water) for the Series N paper has not been accounted for, but may have intruded from lubricants (for the machinery) or from unknown residues in the water system.

4. Pulp fines may carry a disproportionately large fraction of the lithium retained by pulp or paper. Additional experiments with pulp fines may be performed on Project 3101-1.

5. More information about the mineral content of IPC-paper before and after purification will be obtained at the IPC through ashing and emission spectrography.

6. One possible scheme to reduce the amounts of mineral components could involve a device just ahead of the couch roll to displace the process water with deionized water.

7. In addition to conventional papermaking, dry-forming of the sheet may be considered in the development of ultra-low levels of mineral components in filter paper.

8. The application of Kronisol by roll applicator is not suspected as a significant source of lithium.

Introduction

In general accord with the plan as stated in a memorandum dated August 6, 1974, from E. Dickey to Swanson, et. al., I arrived in Watertown on Sunday, August 11, in the late afternoon. Jack Phelps and Sgt. Tom Nibarger, McClellan AFB, MCL-C, arrived after midnight on the same day.

On Monday morning, August 12, at the motel we reviewed the objectives and the plans for the tests to be made in the pilot plant at Knowlton Brothers. An appointment was arranged (by Jack Phelps) for us to meet with Dr. Grant Rauscher at 1:30 p.m. We learned that the pilot plant was being cleaned and readied for the tests which were planned for Tuesday and Wednesday, August 13 and 14.

In the course of our conversations on Monday afternoon, the plans for the tests were reviewed in some detail. It was apparent that the main question to be answered was: "Why is the lithium content of the Series N paper so high?" [The lithium contents of previous lots of the IPC-1478 paper were 4-8 ng Li/g, whereas, Series N paper is approximately 20 ng Li/g.] The pilot plant tests were designed to compare the effects of reprocessed mill water with those of Watertown city water in making the paper. Samples of water collected in October, 1973 during the making of Series N paper analyzed:

ng/ml	city water	mill water after clarification
Lithium	0.8	3.5
Uranium	0.005	0.007

Thus, if the water content of the sheet entering the drier was 75-80%, the city water would have left 3.2-4.0 ng Li/g of paper and the mill water would have left 14-17.5 ng Li/g in the sheet. In the case of the mill water, these amounts could readily account for the final values of ca. 20 ng Li/g in Series N paper.

Pilot Plant Tests

Five tests were planned whereby IPC-1478 paper would be made in the pilot plant as follows:

1. Mill water at pH 9.5; white water recirculated;
2. Mill water at pH 4.1; white water recirculated;
3. City water at pH 9.5; white water discarded;
4. City water at pH 9.5; white water recirculated;
5. City water at pH 9.5; white water recirculated; quantity of paper produced for use at McClellan AFB.

The equipment was thoroughly washed between each test to reduce the cross-contamination of samples. In the first four tests, the run was continued until a steady state had been reached in making paper at 90 lbs/3000 sq. ft., then analytical samples of water and paper were collected and the test was stopped. Upon reaching the correct basis weight in test no. 5, several rolls of paper were collected. This paper will be saturated eventually with Kronisol and used at McClellan.

In the process as used in these tests, 15 lbs. of dry-lapped pulp (Hercules PS-57) was dispersed in a Valley beater to which 80 ml of concentrated ammonium hydroxide (28-29% NH_3, AR) and 25 g. of ammonium carbonate were added. At the headbox, the pulp was diluted with water for a total of 1800 lbs. of water to a consistency of 0.8% at pH 9.5. The sheet was formed

on scrim at 19.3 ft./min. The sheet left the wire at the couch roll (2.5 in. Hg. reduced pressure) and was carried on a felt without pressing to the drier section which consisted of a gas-fired infrared unit followed by eleven 14-inch, steam-heated rolls.

Although the web was pressed at no point, the tension on the web as it passed over the drier rolls determined the final smoothness and density of the sheet. There was some concern that the tension should be maintained at a minimum to prevent any tendency to increase the density of the sheet and thereby reduce its air-permeability. The air-filtration characteristics and particle retention ability will be tested on the Air Force tester (IPC design) housed at Knowlton Brothers.

Possible Sources of Lithium

In reviewing the possible sources of lithium which could raise the lithium content of mill water by factors of four or more over the city water, very few promising ones were found.

In recirculated mill water lithium may be leached from the concrete floor underneath the machine and from the piping, but these are considered to be a minor source.

Because the lithium salts of fatty acids are known to be added to some lubricants, samples of oil and grease were obtained for lithium analysis. Other real sources, if any, remain hidden. If the lithium contents of the several samples from the pilot plant tests are inexplicably high, an intensive search will be started to find the source of the lithium.

Possible Role of Fines

One of the tests performed by Capt. Grosso involved the white water from the Series N papermaking operation in October, 1973. A sample of the water was filtered through pads of L-series paper (4.2 ng Li/g), and the paper was dried and analyzed. The lithium contents were then 34-70 ng Li/g. These results together with the results of an experiment on Project 3101 (Report Ten, p. 12) support the probability that pulp fines concentrate the lithium. To understand better the possible role of fines in the retention of lithium in IPC-1478 paper, further experiments are being considered. A closely related experiment will attempt to determine whether or not the ion-exchange capacity of the pulp may be sufficient to remove lithium from process water. Previous experiments with lithium-6 (Report Seven, p. 16) indicated that most, if not all, lithium may be exchangeable.

Other Factors Affecting the Lithium Content

IPC-1478 filter paper, Series-L, was made from pulp which had been specially washed by Hercules, Inc., and the lithium and uranium contents of the L-series were the lowest of any of the lots made by Knowlton Brothers. In addition to the use of specially purified pulp, the process water was taken from the Black River instead of the Watertown city water as was the case for the Series-N paper. Also, the process water was filtered through anthracite and diatomaceous earth and the white water was not recycled. The use of the anthracite and diatomaceous earth filters has been discontinued, and the white water for the Series-N paper was recycled.

Although these differences in the manufacturing processes for Series-L and Series-N may contribute to the markedly higher level of lithium in the Series-N paper, the main source of the lithium remains hidden.

Filtration and Retention Tests

IPC-1478 paper, Series-N, had been purified on Project 3101, treated with Kronisol, and sent to McClellan AFB as sample 3101-350 (see Report Thirteen, p. 5). Several specimens of this lot of paper were subsequently sent to Knowlton Brothers by the McClellan Laboratory for air filtration and retention tests. The Laboratory at Knowlton Brothers applied Kronisol to the specimens before testing without knowing that Kronisol had been added at the IPC. The results are listed on the attached data sheet. As interpreted by Dr. Rauscher and Jack Phelps, the performance showed that the purification process had not changed the required properties of the paper.

Ash Content and Composition cf IPC-1478 Paper

The analytical procedure used by McClellan AFB, MCL-C includes no measure of total ash. Consistent with tentative plans outlined previously (see Memo dated June 21, 1974, Dickey to Swanson, et al.) several types of paper, purified and unpurified, will be submitted to the IPC Analytical Group for (a) total ash and (b) elemental analysis by emission spectrography. One of the questions which may be answered in these studies is concerned with the brown color of the ash from Series-N paper in contrast to the ashes of other papers which are white.

Miscellaneous Observations and Comments

1. One run of paper on the pilot plant was made at pH 4.1 (measured at the headbox) obtained by the addition of hydrochloric acid. The pH was chosen to test the effect of acid on the retention of lithium by the paper. The performance apparently was the same as at pH 9.5 except that the white water was essentially free of fines. This effect was unexpected and may signal other differences in the properties of the sheet.

2. Because of the sensitivity of the uranium and lithium contents of IPC-1478 paper to water quality, Dr. Rauscher suggested that forming the sheet in air instead of water should favor the production of paper with low levels of impurities. Such methods have been considered in past discussion and rejected because of the major effort required to form and bond a satisfactory sheet. However, the possibilities for the production of a satisfactory filter paper with very low levels of impurities by an air-forming process are real and may appear eventually more practical than at present.

3. The crew on the pilot plant consisted of Paul Nutting, pulpman; Bob Campaney, machine tender; and Don DeForde, pilot machine supervisor. Dr. Grant Rauscher was responsible for planning the work and the main course of the tests. The crew worked together very effectively.

4. In a conversation with Dr. Rauscher we raised the possibility of installing a device just before the couch roll which would preserve the formation and provide sufficient deionized water to displace the process water from the sheet as it passed over the couch roll. Such a device should remove most of the dissolved impurities which would otherwise be dried on the paper. The sheet passes to the drier section at approximately 76% water so that process water at 4 ng. Li/ml. alone would carry 12 ng. Li/g into the finished sheet. A quick washing step with "pure" water at the couch roll should remove most of this load of lithium.

5. Kronisol is applied by a roll applicator (saturator). The machine is used for many types of specialty papers with exposure to many types of chemical materials. However, the equipment appears to be cleaned readily and is probably not significant in adding impurities to the finished IPC-1478 paper. Kronisol, itself, is usually low-to-insignificant in its lithium content.

The results of these tests should provide at least a partial answer to the question, "Why is the lithium content of Series-N IPC-1478 paper abnormally high?"

EED/ctb
Attachment

GRADE 107 IPC # 3101-350 IPC 1-18 POST-TREATED SAMPLES K-24% KRONISOL

8/9/74

SAMPLE NO. OR LOT NO.	PRESSURE DROP INCHES OF WATER LEFT MAN 1.25" ORIFICE	AIR VELOCITY FT/MIN. THRU SPECIMEN	PRESSURE DROP INCHES OF WATER ACROSS SPECIMEN RIGHT MAN.	UPPER CELL CLEAN AIR * A	UPPER CELL DUST LADEN AIR NO SPECIMEN B	LOWER CELL CLEAN AIR * C	LOWER CELL DUST LADEN AIR NO SPECIMEN E	UPPER CELL DUST LADEN AIR WITH SPECIMEN G	LOWER CELL DUST LADEN AIR WITH SPECIMEN F	E / RETENTION %
K 1	40	2150	30	32	28	32	28	92	34	96.9
K 3	36	2040		32		32			34	96.9
7	38	2100		33		32			34	96.9
K 8	33	1950		33		33			35	96.9
9	40	2150		33		33			35	96.9
K 10	36	2040		32		33			36	96.9
13	42	2200		33		33			35	96.9
K 18	40	2150.		32		33			35	96.9

* Due to contamination of air, paper No. IPC 1478 (or equivalent) should be inserted in sample holder.

APPENDIX III

AIR TEST DATA FOR SELECTED SAMPLES
No. 3101- 365, 366, 376, 378, 426, 409, 425, 428, 430

These tests were performed on the USAF tester at Knowlton Brothers, Inc., Watertown, NY, and reported by letter dated February 24, 1975, from Dr. Grant K. Rauscher to E. E. Dickey and to Jack Phelps.

Department of the Air Force
1155th Technical Operations Squadron (Hq. Comd.)
McClellan AFB, California
Project 3101-1

- 1478 Paper (KB 107) IPC Series 3101

SAMPLE NO. OR LOT NO.	PRESSURE DROP INCHES OF WATER LEFT MAN 1.25" ORIFICE	AIR VELOCITY FT/MIN. THRU SPECIMEN	PRESSURE DROP INCHES OF WATER ACROSS SPECIMEN RIGHT MAN.	UPPER CELL CLEAN AIR * A	UPPER CELL DUST LADEN AIR NO SPECIMEN B	LOWER CELL CLEAN AIR * C	LOWER CELL DUST LADEN AIR NO SPECIMEN E	UPPER CELL DUST LADEN AIR WITH SPECIMEN G	LOWER CELL DUST LADEN AIR WITH SPECIMEN F	RETENTION %
365	4.8	2350	30	35	98	35	98	98	38	95.2
366	4.3	2225		30		35			38	95.2
376	4.7	2325		28		35			37	96.8
378	4.4	2250		28		35			37	96.8
426	5.6	2550		29		35			38	95.2
426	4.7	2325		33		36			38	95.2
426	4.1	2175		30		36			38	96.8
409	4.3	2225		33		36			39	95.2
409	5.1	2425		33		37			39	96.7
409	5.8	2600		31		36			39	95.2
425	5.3	2475		33		36			39	95.2
425	5.3	2475		31		36			39	95.2
425	4.8	2350		32		37			39	95.1

Continued Page -2-

* Due to contamination of air, paper No. IPC 1478 (or equivalent) should be inserted in sample holder.

IPC - (KB 107) IPC Series 3101

SAMPLE NO. OR LOT NO.	PRESSURE DROP INCHES OF WATER LEFT MAN 1.25" ORIFICE	AIR VELOCITY FT/MIN. THRU SPECIMEN	PRESSURE DROP INCHES OF WATER ACROSS SPECIMEN RIGHT MAN.	UPPER CELL CLEAN AIR * A	UPPER CELL DUST LADEN AIR NO SPECIMEN B	LOWER CELL CLEAN AIR * C	LOWER CELL DUST LADEN AIR NO SPECIMEN E	UPPER CELL DUST LADEN AIR WITH SPECIMEN G	LOWER CELL DUST LADEN AIR WITH SPECIMEN F	RETENTION %
428	4.7	2325	30	32	98	37	98	98	39	96.7
428	5.1	2425		31		37			40	95.1
428	4.6	2300		32		37			40	95.1
430	4.7	2325		29		37			40	95.1
430	4.6	2300		31		37			40	95.1
430	4.6	2300		35		37			39	96.7
430	5.0	2400		35		38			40	96.7

* Due to contamination of air, paper No. IPC 1478 (or equivalent) should be inserted in sample holder.

Department of the Air Force
1155th Technical Operations Squadron (Hq. Comd.)
McClellan AFB, California
Project 3101-1

APPENDIX IV

PROJECT 3101-1 SAMPLES LISTED IN CONSECUTIVE ORDER

Location of analytical data by table; surplus samples

For Samples 148 to 364 see Report Fourteen, Appendix III, p. 79

Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.	Sample No.	Table No.
365	II	384	III	410	II	436	V	462	VI
366	II	385	III	411	I	437	V	463	a
367	I	386	II	412	V	438	III	464	VI
368	II	387	a	413	V	439	III	465	VI
369	I	388	a	414	V	440	a	466	a
370	II	389	II	415	a	441	III	467	a
371	I	390	I,IV	416	a	442	a	468	VI
372	II	391	a	417	a	443	III	469	VI
373	I	392	II	418	a	444	III	470	II
374	II	393	IV	419	a	445	a	471	I
375	I	394	IV	420	a	446	a	472	II
376	II	395	I	421	V	447	a	473	II
377	I	396	IV	422	V	448	III	474	II
378	II	397	IV	423	II	449	a	475	II
379	I	398	II	424	I	450	a	476	II
380	a	399	III	425	II	451	III	477	II
381	a	400	a	426	II	452	a	478	I
382	I	401	III	427	II	453	III	479	a
383	III	402	III	428	II	454	III	480	VII
		403	IV	429	II	455	a	481	a
		404	IV	430	II	456	a	482	VII
		405	IV	431	a	457	VI	483	II
		406	II	432	a	458	VI	484	II
		407	IV	433	a	459	a	485	II
		408	I	434	a	460	a	486	II
		409	II	435	V	461	VI	487	I
								488[b]	II

[a] Surplus samples; these are retained at the Institute for one year and are available on request.

[b] Final number assigned on Project 3101-1.