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Removal of Organic Contaminants From Aluminum Chloride Solutions

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UNITED STATES DEPARTMENT OF THE INTERIOR

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REMOVAL OF ORGANIC CONTAMINANTS FROM ALUMINUM CHLORIDE SOLUTIONS

by

Jack C. White,[1] Jack L. Henry,[2] and Charles J. Krogh[3]

ABSTRACT

The Bureau of Mines, in its efforts to insure the continued viability of the domestic minerals economy, has been engaged for several years in research on the extraction of alumina from domestic, nonbauxitic resources. Hydrochloric acid leaching of kaolin is one of the more promising technologies being studied for recovering alumina for feedstock to existing aluminum smelting capacity; 93 pct of this feedstock is currently imported either as bauxite or alumina.

Organics contamination of pregnant leach liquor, originating during solvent extraction for removal of iron from aluminum chloride solution formed during hydrochloric acid leaching, causes rapid deterioration of the semihard rubber lining of process equipment. These organics were removed by a skimming tank-coalescer-activated carbon adsorption system. Organic levels of less than 10 ppm from the coalescer and less than 0.5 ppm from carbon adsorption were achieved.

Analytical methods employing CCl_4 extraction and infrared spectrophotometry were developed to determine the organic content of both the liquor and the carbon.

Mass transfer zones delineated in the carbon columns provide adsorption data for the design of larger systems.

INTRODUCTION

The mission of the Bureau of Mines is to help insure the continued viability of the domestic minerals economy and the maintenance of an adequate minerals base so that the Nation's economic, social, strategic, and environmental needs can be better served.

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Research has been conducted by the Bureau for several years to develop technology for extracting alumina from domestic nonbauxitic resources. This program is in harmony with the goal of insuring a dependable domestic supply of minerals and mineral-based materials adequate to meet the Nation's industrial, economic, and security needs.

Several technologies for extraction of alumina are being examined by the Bureau. The hydrochloric acid (HCl) leaching of kaolin is under study in a miniplant program at the Boulder City Engineering Laboratory. In this technology, calcined kaolinitic clay is digested in HCl to produce an impure aluminum chloride ($AlCl_3$) leach liquor from which iron is removed by solvent extraction. The $AlCl_3$ is crystallized from solution by sparging HCl gas into the pregnant liquor with subsequent thermal decomposition of crystalline $AlCl_3$ hexahydrate to alumina and HCl.

Experiments have shown that semihard natural rubber is resistant to the corrosive action of leach solutions that contain HCl concentrations as high as 27 pct at temperatures up to 85° C. Tests also indicated that residual organic[4] contamination of the pregnant liquor, originating in the solvent extraction unit, attacks and destroys natural rubber. Natural rubber-lined equipment, therefore, must not be exposed to liquor containing organics. Removal of organics from $AlCl_3$ liquor is the subject of this report.

The organic phase in the solvent extraction unit consisted of 75 vol-pct kerosine, 15 vol-pct Alamine 336,[5] and 10 vol-pct isodecanol (commercial-grade mixed isomers of decyl alcohol). The organic phase, used in mixer-settlers to remove iron from pregnant liquor, becomes entrained and dissolved to a greater or lesser extent in the $AlCl_3$ process stream. Gross organic contamination should be removed by means of skimming tanks and coalescers so that the organics may be recovered for reuse. The remaining few parts per million of entrained and dissolved organics may be removed by adsorption on fixed-bed granular activated carbon or by batch contacting with powdered activated carbon. A premium-grade, activated, coconut hull carbon was used in this study. Carbon columns were designed by utilizing the mass-transfer-zone concept[6] wherein flow rate and composition of the input and output streams are measured, and the resulting length, position, and rate of migration of the mass transfer zone (MTZ) are determined by analysis of the carbon bed. Engineering data developed in this manner can be used to design larger systems with confidence. This approach to the initial design of a carbon column for removing organics from $AlCl_3$ liquor eliminated the need for laboratory determination of adsorption isotherms, a procedure that proved unreliable because of difficulty in producing standardized samples of liquor containing entrained

[4]The terms "organic" or "organics" as used in this report refer to the mixture of organic solvents, extractants, and phase modifiers used for purification of the $AlCl_3$ liquor. These terms are not applied to CCl_4 used in analytical determinations.

[5]Reference to specific trade names or manufacturers does not imply endorsement by the Bureau of Mines.

[6]Luchis, G. M. Adsorption Systems. Part 1: Design by Mass-Transfer-Zone Concept. Chem. Eng., v. 80, No. 13, June 11, 1973, pp. 111-118.

organics and inadequate sensitivity of analysis for organics during early stages of this work.

Sensitive methods were developed for the determination of organics in both the pregnant liquor and the carbon adsorbent. The method involved the extraction of organics from the liquor and from the spent carbon with high-purity carbon tetrachloride (CCl_4). The organics in the extraction solvent were then determined by infrared (IR) spectrophotometry.

EXPERIMENTATION

Physical Condition of Organics in Liquor

Organic contamination of $AlCl_3$ liquor may be in the form of a separate phase, either floating or suspended (emulsified), in addition to the dissolved organics. Physically entrained organics are amenable to recovery by physical means, such as skimming tanks that remove gross contamination and coalescers that consolidate dispersed organic phase into reusable form. To remove dissolved organics from the $AlCl_3$ liquor, the technique, commonly employed in industrial processes of adsorption on activated carbon in a fixed bed was chosen.

Carbon Column Design

The fixed-bed, activated carbon columns were designed and operated in accordance with acceptable industrial practice. Because adequate design data were not available, the following four assumptions were made relating to design of the columns: (1) that $AlCl_3$ would not interfere with the adsorption of organics on carbon, (2) that liquor would contain 50 ppm organics, (3) that 2 grams of organics per 100 grams of carbon was a conservative loading figure, and (4) that the mass transfer zone would be less than 6 feet long. Design criteria are as follows:

Pregnant liquor:	
Volume..........gal..	3,000
Density..........g/ml..	1.23
Flow rate..........gal/hr..	25.5
Organics content..........ppm..	50
Carbon type..........	coconut charcoal
Carbon particle size..........mesh..	12-20
Organics loading on carbon..........wt-pct..	2.0
Column diameter..........inches, ID..	4.0
Carbon weight..........lb/ft of column..	2.30

The calculated column design criteria are as follows:

Pregnant liquor:	
Weight..........lb..	30,794
Velocity in unpacked column..........in/min..	7.8
Organics weight..........lb..	1.54
Carbon required..........lb..	76.9
Carbon bed depth..........ft..	33.4
Bed expansion factor for elutriation..........	1.5
Total column length..........	50.1

A 10-foot length of 4-inch pipe will hold a 6.7-foot bed capable of treating at least 600 gal of liquor according to the above assumptions and design criteria.

Sampling of Liquor and Carbon

Liquor samples taken during the miniplant operation were poured directly from flowing liquor streams into sealable glass flasks. Exceptional care in sampling the liquor was necessary because the walls of any intermediate container used in sampling will act as a coalescer, removing suspended organic droplets from the aqueous phase.

Carbon columns were displacement washed with downward flowing, slightly acidified water (pH 2.5) to remove $AlCl_3$ prior to sampling. Samples were taken by slicing 2-inch segments from the carbon bed as it was pushed out of the glass pipe. The wet carbon samples were shipped in sealed jars to prevent loss of volatile organics.

Development of Analytical Methods

Analysis of the organics content of purified pregnant $AlCl_3$ liquor was necessary to determine the effectiveness of organics removal by means of activated carbon adsorption, and analysis of the activated carbon adsorbent was necessary to determine carbon consumption and the configuration of the mass transfer zone. Methods developed for the analysis of organics in liquor and in carbon were based on the high sensitivity attainable by infrared absorption at the C-H stretch frequency of 2,924 cm^{-1}. Organics dissolved in spectrographic-grade CCl_4 (which contains no measurable C-H bonds) were determined by recording the absorption spectrum of the organics-CCl_4 solutions through the C-H stretch frequency by means of an IR spectrophotometer. This method is relatively insensitive to differences among kerosine, Alamine 336, and decanol; therefore, individual compounds cannot be easily identified.

Standard solutions of the mixed organics used to extract iron from $AlCl_3$ leach liquors were made by successive dilution with spectrographic grade CCl_4. Presumably, these solutions were of the same composition as organics entrained in $AlCl_3$ liquor. The standard solutions, analyzed by IR spectrophotometry, provided a calibration curve described by a second-order regression equation (fig. 1). The organics content of unknown solutions were calculated from the regression equation.

Analysis For Organics in Purified Pregnant $AlCl_3$ Liquor

The kinetics of extraction of organics dissolved in purified pregnant $AlCl_3$ liquor was investigated to determine the required liquor-CCl_4 mixing time. Dissolved organics were extracted with CCl_4 by shaking in separatory funnels in a specially built reciprocating machine. Extraction was essentially complete within 2 min; however, all analytical extractions involved a 10 min mixing time (fig. 2). Presumably, physically entrained organics would be quickly taken into solution.

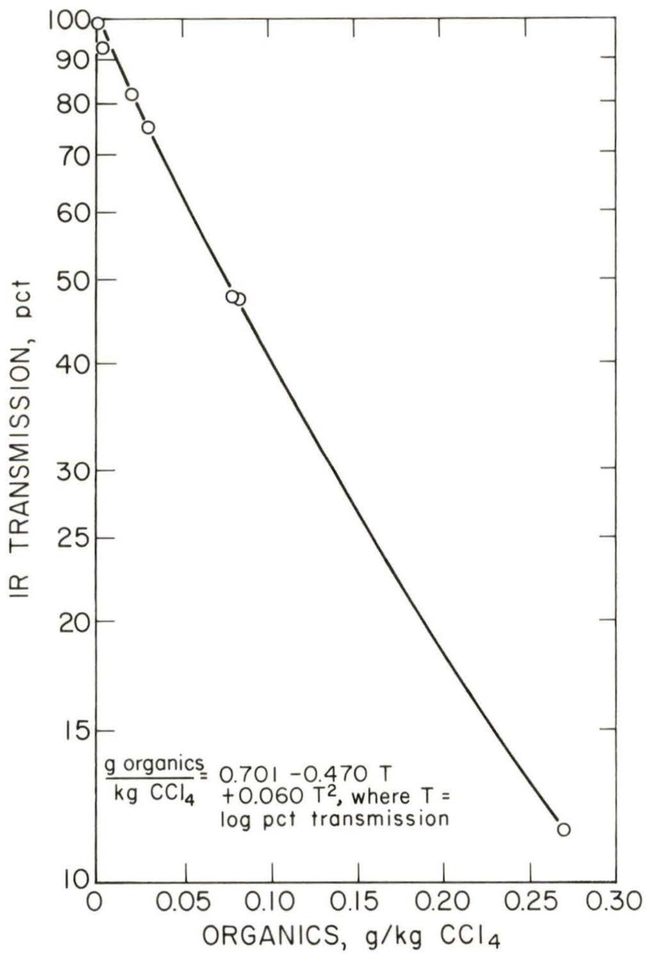

FIGURE 1. - Calibration curve for infrared determination of organic content of CCl_4. C-H stretch frequency of 2,924 cm^{-1}.

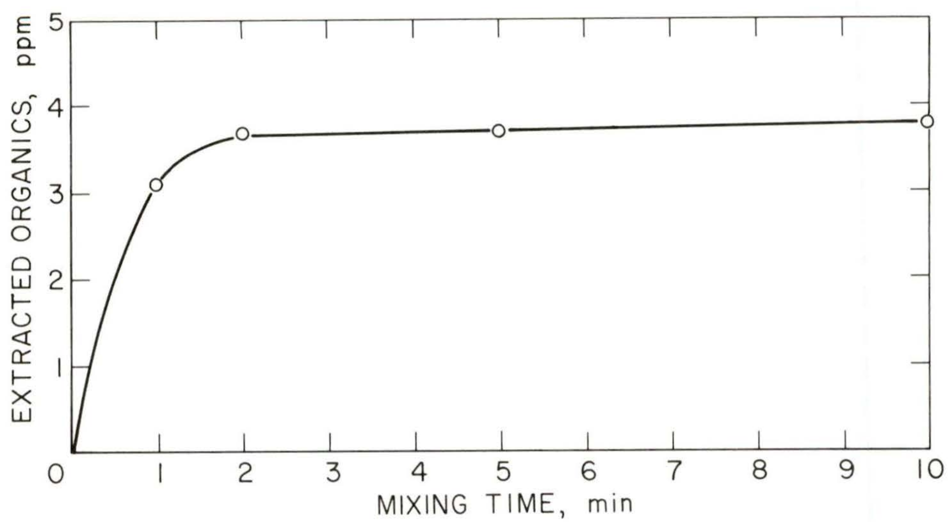

FIGURE 2. - Extraction of organics from $AlCl_3$ liquor using CCl_4.

During analysis of unknown liquors, organics were extracted with three successive equal portions of CCl_4 by shaking in separatory funnels in the reciprocating machine. Phase disengagement was difficult at room temperature, but was more easily accomplished while separatory funnels were immersed in a 40° C water bath. The CCl_4 portions were composited prior to IR analysis. Care was taken during analysis to extract the organic coating on the walls of the shipping containers.

A second calibration curve illustrates a systematic relationship between IR-measured organics and the organics content of doped liquor. The IR-measured values were higher than the true values by about 15 pct; therefore, all measured values were corrected by a derived factor of 0.8534 (fig. 3). Corrections were necessary in part because of vaporization of CCl_4 and other losses that prevented exact determinations of CCl_4 weights.

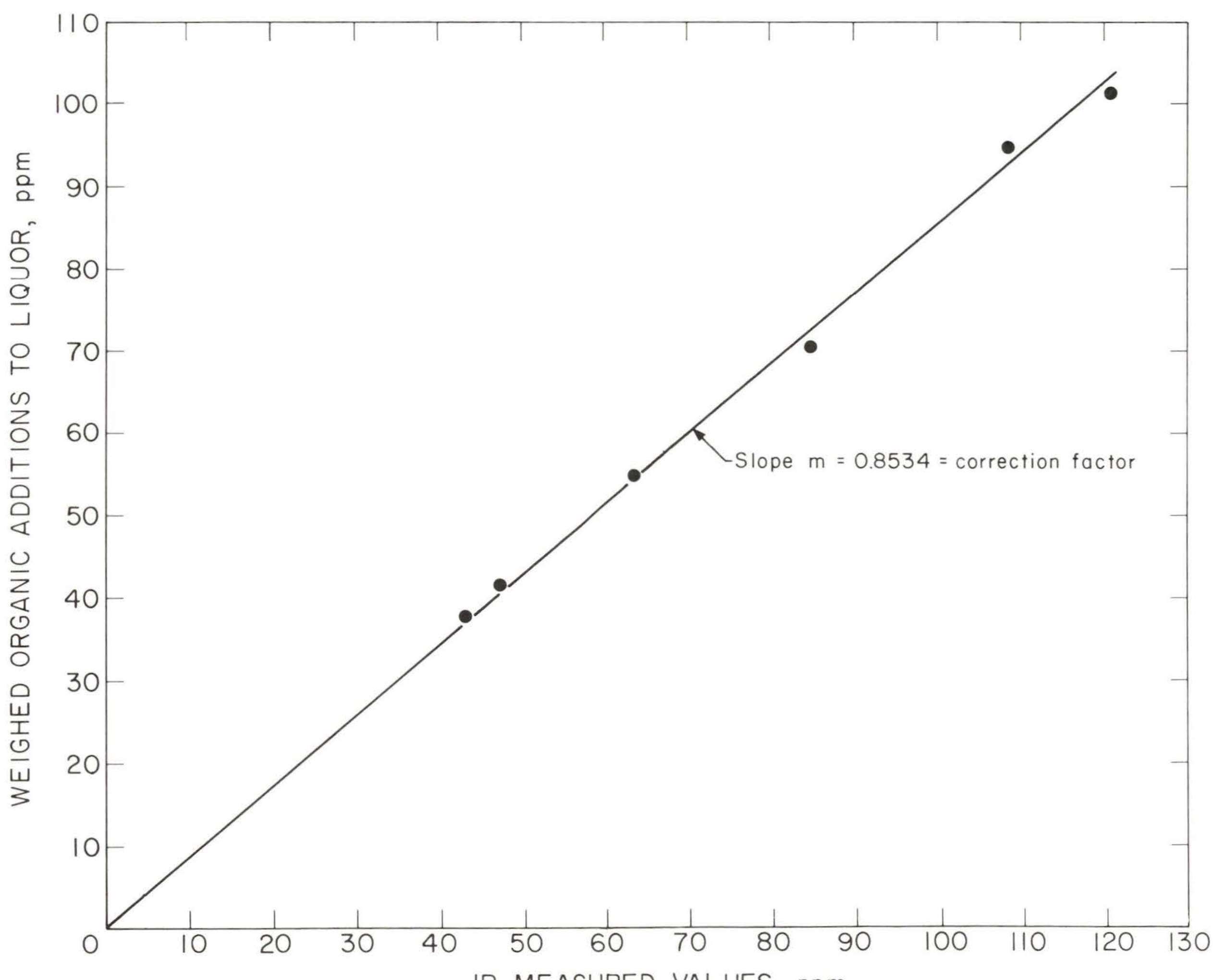

FIGURE 3. - Correlation between weighed organics added to liquor and IR-measured values (organics). Organics additions include 4 ppm dissolved organics.

Analysis for Organics in Activated Carbon Adsorbent

Wet carbon samples were air dried prior to CCl_4 extraction. Room-temperature air drying minimized loss of potentially volatile organics. Organics were extracted from carbon samples with CCl_4 in a Soxhlet extraction apparatus and subsequently determined by IR spectrophotometry.

Kjeldahl analysis of carbon both before and after CCl_4 extraction provided information on its organic nitrogen (amine) content and on amine extraction by CCl_4. Nonreproducibility of nitrogen analyses and the small amount present indicates general trends in amine adsorption, but should not be regarded as a quantitatively accurate analysis of amines present in carbon adsorbent.

Details of the analytical procedures are presented in appendix A; data and sample calculations are presented in appendix B.

RESULTS

The solubility of organics in purified pregnant liquor at room temperatures was found to be 4 ppm. This value was approached asymptotically as shown by the extraction kinetics curve (fig. 2). Liquor samples used for these analyses were withdrawn from the center of a barrel of purified pregnant liquor that had been undisturbed for at least 6 months. Care was taken to avoid contaminating the samples with the separated organic phase floating on the liquor surface. Presumbly, the liquor was in equilibrium with organics and contained no suspended (emulsified) organics.

Organics, both entrained and dissolved, were effectively removed from purified pregnant liquor by the skimming tank-coalescer-carbon column train. Input (polished pregnant liquor) to the solvent extraction unit was low in organics (0.3 to 1.2 ppm), and the output either from carbon columns or from powdered charcoal batch treatment was very low in organics (0.2 to 0.35 ppm); therefore, it is apparent that organic contamination of liquors originates predominantly in the solvent extraction unit. Intermediate process streams were variable in organic content. The following data, taken from a typical run, exemplify the variability in organics content of intermediate process streams:

	Parts per million
Coalescer:	
Feed....................	314
Discharge...............	6.2
Carbon column:	
Feed....................	5.6
Discharge...............	.3

During this run, the coalescer effectively reduced organics content of liquor almost to the solubility limit of 4 ppm. The organics content of the various process streams are presented in table 1.

TABLE 1. - Liquor analyses (organics content), parts per million

Sample and date (1977)	1 (4/21)	2 (4/26)	3 (4/27)	4 (4/28)	5 (5/6)
Pregnant liquor.........	0.60	0.48	1.2	0.31	ND
Purified pregnant liquor	97	67	ND	ND	82
Coalescer feed..........	ND	178	ND	71	265
Coalescer discharge.....	ND	56	ND	12	ND
Liquor to carbon adsorbents.............	ND	3.9	ND	ND	8.2
Liquor from carbon adsorbents.............	ND	.20	ND	ND	.24
Strip liquor............	24	19	ND	ND	ND
	6 (5/6)	7 (5/6)	8 (5/9)	9 (5/11)	10 (5/13)
Pregnant liquor.........	ND	ND	ND	ND	ND
Purified pregnant liquor	38	ND	ND	ND	ND
Coalescer feed..........	314	ND	ND	ND	ND
Coalescer discharge.....	6.2	ND	ND	ND	ND
Liquor to carbon adsorbents.............	5.6	5.0	3.6	2.5	ND
Liquor from carbon adsorbents.............	.30	.35	ND	.21	.20
Strip liquor............	ND	ND	ND	ND	ND

ND No data.

Both activated granular carbon columns and batch treatment with powdered carbon reduced the organic content of purified pregnant liquor to less than 0.5 ppm, demonstrating that dissolved organics can be effectively adsorbed from $AlCl_3$ liquors. Because both methods were effective, a choice between them may be based on cost of operation and efficiency of carbon utilization. Both factors appear to favor carbon columns.

Short mass transfer zones in the three carbon columns demonstrate that adsorption of organics was rapid. Most of the organics were removed from liquor within a mass-transfer zone length of 3 inches or less. Liquor velocity of 7.8 in/min in unpacked bed would be 15.6 in/min in a bed packed with 50 vol-pct carbon. Thus, 3 in/15.6 in min^{-1} equals the adsorption time of 0.19 min (11.5 sec).

Organic loading curves were not as well developed as was hoped. Ordinarily, columns develop three well-defined zones, an equilibrium zone where saturated carbon is in equilibrium with loaded liquor, a mass transfer zone where adsorption occurs, and the unused bed. Where these zones are well developed, the position of the "stoichiometric front" is easily determined, and from this position the total capacity of a column may be calculated. Two problems were encountered in precisely locating the stoichiometric front in these columns. First, an equilibrium zone had not developed; and secondly, the feed stream contains both dissolved and physically entrained organics, whereas the equilibrium zone concept is based only upon soluble substances. For this reason, precise location of the stoichiometric front may be difficult even after columns have been run for longer periods of time. Estimates of total column capacity were made by drawing the position of stoichiometric fronts (dotted vertical lines) through the steepest portions of the organic loading curves, and calculating column capacity accordingly (figs. 4-6). This method, although not entirely rigorous, should provide data adequate for scale-up to demonstration plant size where more precise data can be determined.

Organic nitrogen compounds, presumably amines from Alamine 336, penetrate farthest down the carbon columns as determined by Kjeldahl analysis of carbon adsorbent (figs. 7-9). Although the quantity of amines penetrating down the columns is relatively small, they potentially may cause the greatest difficulty in removal of organics from $AlCl_3$ liquor. However, the effect of these amines on rubber linings was not determined. The best protection for rubber linings probably would be provided by maintaining several feet of unused carbon column downstream from the mass transfer zone.

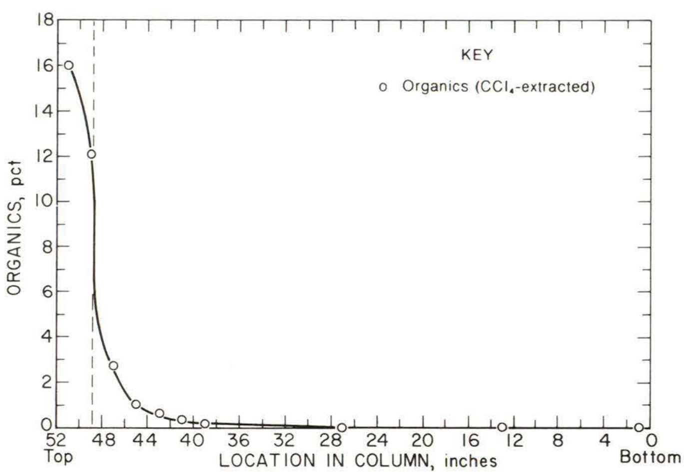

FIGURE 4. - Organics loading in column 1.

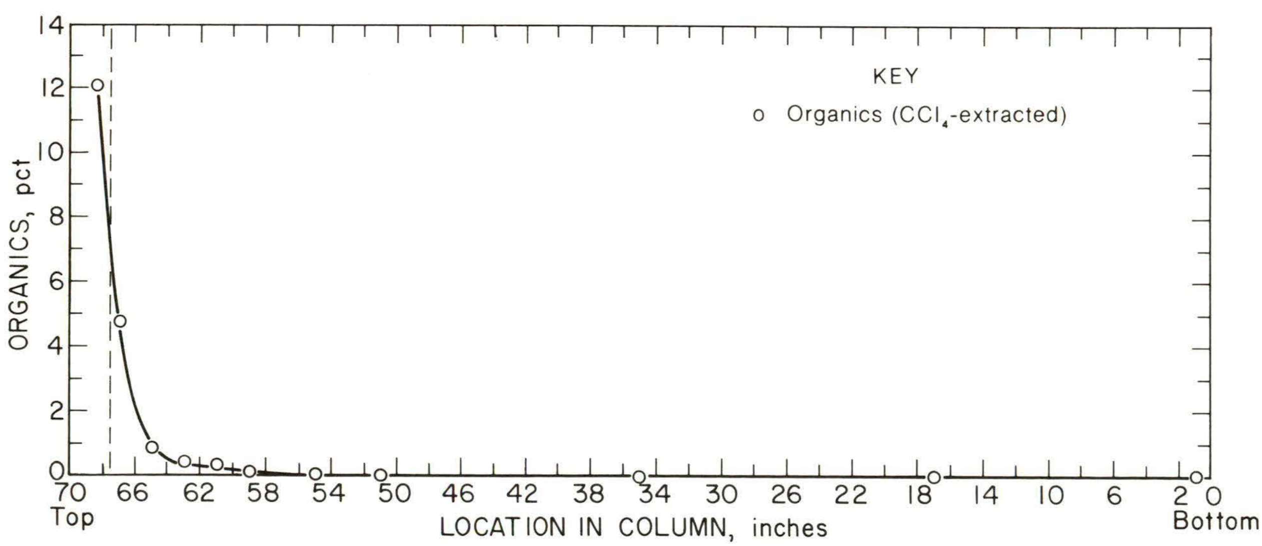

FIGURE 5. - Organics loading in column 2.

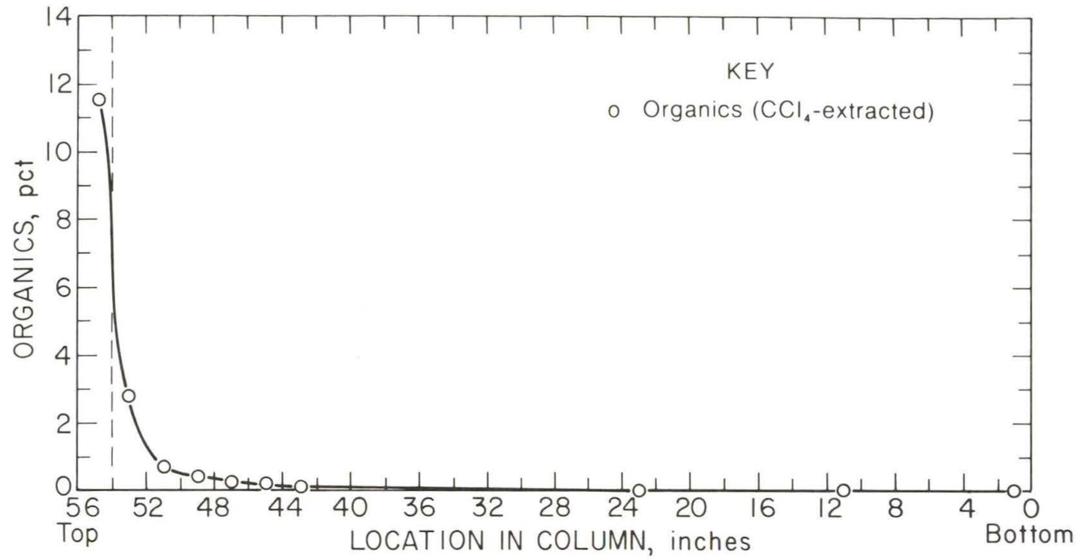

FIGURE 6. - Organics loading in column 3.

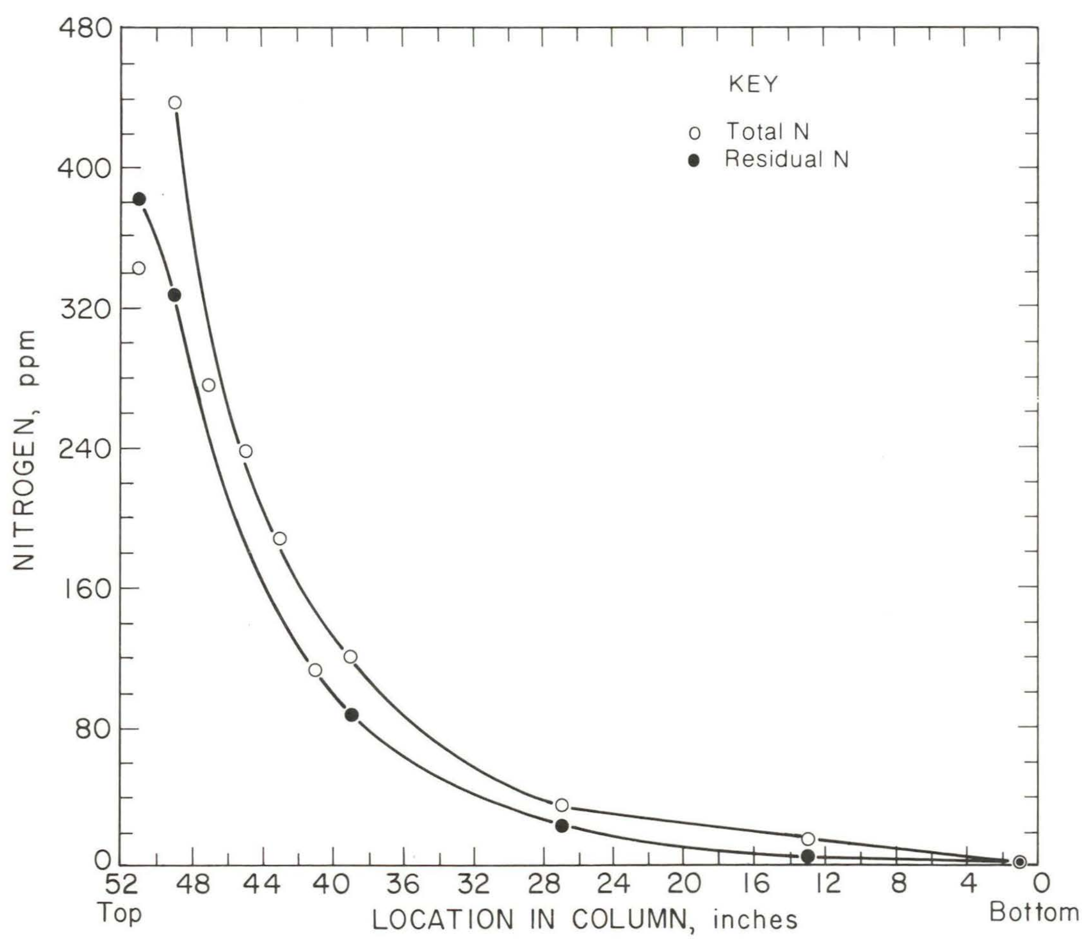

FIGURE 7. - Organic nitrogen profile for column 1.

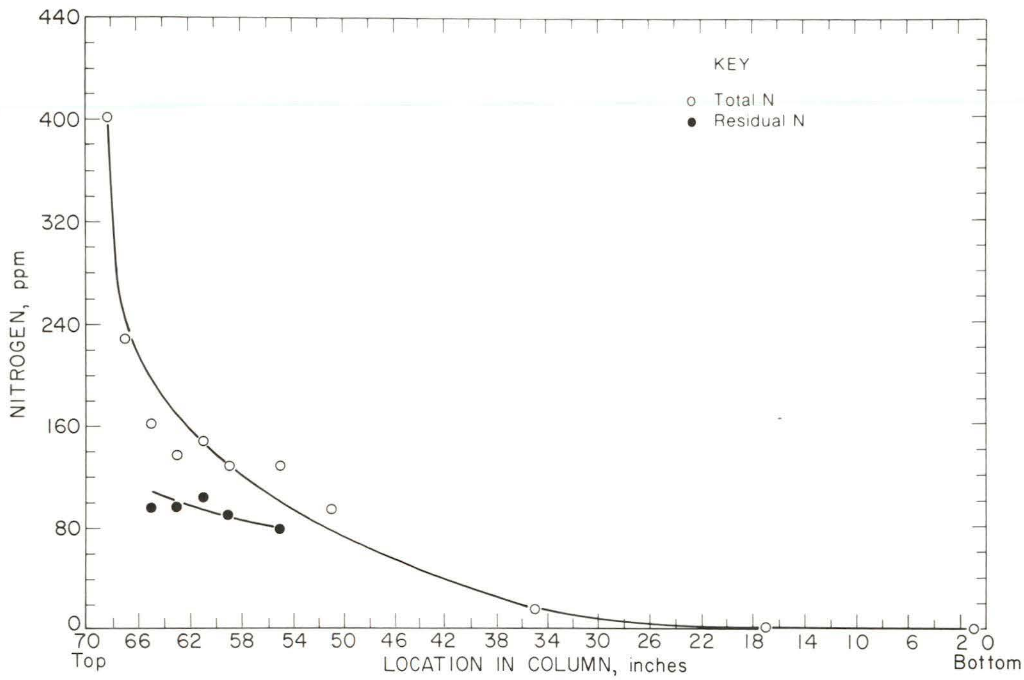

FIGURE 8. - Organic nitrogen profile for column 2.

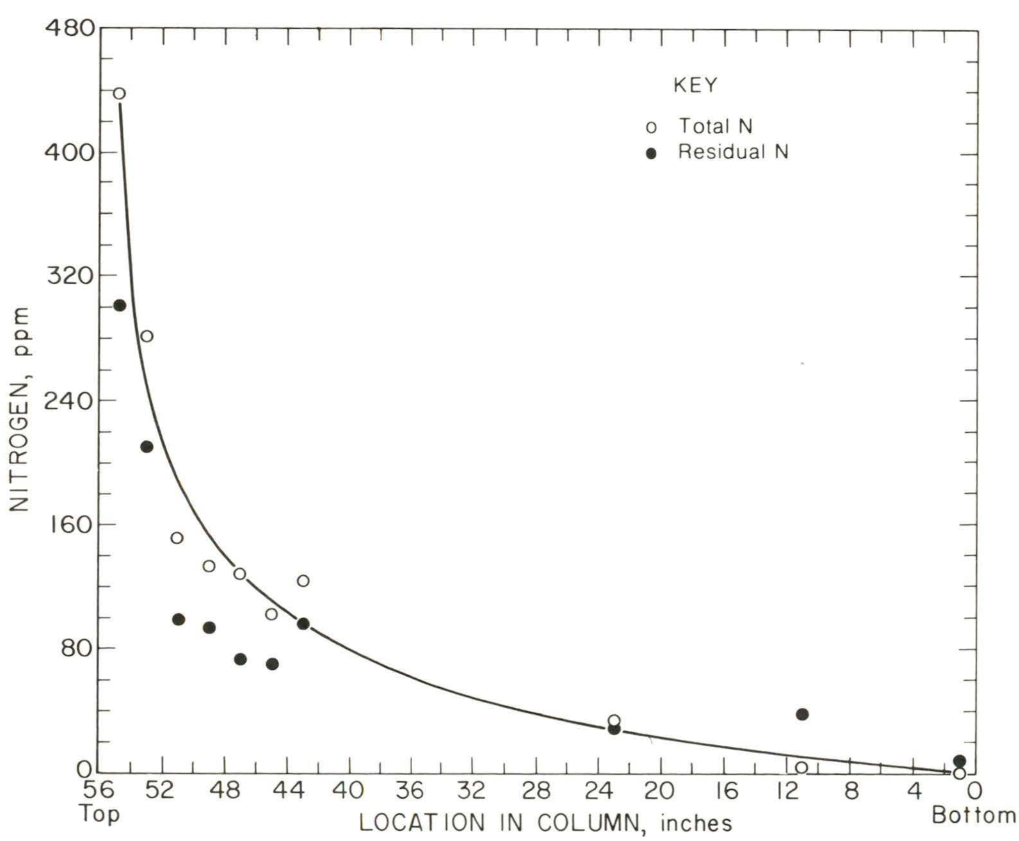

FIGURE 9. - Organic nitrogen profile for column 3.

CONCLUSIONS

1. CCl_4 extraction-IR spectrophotometry is a satisfactory method for determining the organic content of $AlCl_3$ liquor and carbon adsorbent.

2. The solubility limit of organics in pregnant $AlCl_3$ liquor at room temperature is approximately 4 ppm.

3. The coalescer reduced the organic content of liquor to 6 ppm, which closely approaches the solubility limit of 4 ppm.

4. Granular activated carbon columns reduced organics to less than 0.5 ppm, demonstrating that carbon adsorption removes dissolved organics from $AlCl_3$ liquors.

5. Water-soluble amines penetrate farther down carbon columns than CCl_4-extractable organics. The potential threat of amine breakthrough causing destruction of rubber linings must be considered in the design of a larger system. The effects of the amine component of the organics on rubber linings was not determined.

6. Rubber linings probably will not be adversely affected by the small content of residual organics present in carbon treated $AlCl_3$ liquor.

RECOMMENDATIONS

1. Organic contaminated $AlCl_3$ liquors should be treated with an equipment train consisting of a skimming tank-coalescer-granular activated carbon column to remove organics prior to further processing of the liquors in rubber-lined equipment.

2. Adsorption of organics should be conducted in two or three columns in series, always maintaining 4 to 5 feet of unused bed downstream from the major mass transfer zone to prevent breakout of the water-soluble organics.

3. Liquor and/or carbon must be analyzed periodically as a control on the adsorption process.

4. Larger scale and longer periods of operation will be required to firmly establish carbon consumption.

APPENDIX A.--CALIBRATION DATA

The organics-in-CCl_4 calibration curve (fig. 1) was produced by adding weighed amounts of organics to CCl_4 and by making suitable dilutions based on weights. The standard samples were scanned in an IR grating spectrophotometer to determine maximum absorption at the C-H bond stretch wavelength of 2,924 cm^{-1}. Plotting log of percent IR transmission versus the concentration of organics provided a suitable calibration curve. An nth order regression equation, determined from calibrating data, was used in calculating organic concentrations of all unknown solutions (table A-1; sample calculations, appendix B).

TABLE A-1. - Standard samples used in calibration of infrared grating spectrophotometer

Organics,[1] g/kg CCl_4	IR transmission, pct	Log pct transmission
0.2700	11.5	1.0607
.0820	47.5	1.6767
.0790	48.0	1.6812
.0301	75.5	1.8799
.0205	82.5	1.9165
.0078	93.0	1.9685
.0000	100.0	2.0000

[1]Dissolved in CCl_4.

A final correction curve was made by adding weighed amounts of organics to purified pregnant liquor that contained 4 ppm of dissolved organics, extracting the doped liquor with CCl_4 by the standard method, and analyzing by IR spectrophotometry. IR-measured organics were consistently higher by a few percent than the amount of organics added. Thus, the need for a correction curve became apparent. The systematic error probably is due to vaporization loss of CCl_4 during extraction and to other inaccuracies in determining the exact weight of CCl_4.

Presumably, the same correction was necessary when analyzing very low values, 0 to 5 ppm of organics, but standards in this range could not be made accurately. Because (0, 0) is a legitimate data point, the regression line was forced through the origin by entering a negative set of the (x, y) values with the positive values to provide a regression line that includes (0, 0). This approach increased standard deviating slightly, but made possible the correction of values less than a few parts per million (table A-2; sample calculations in appendix B).

TABLE A-2. - Standard samples of organics in liquor used in determining the correction factor, parts per million

Organics added to $AlCl_3$ liquor	Organics extracted by CCl_4 and measured by IR spectrophotometry
37.81	43.00
41.46	47.23
54.76	63.48
70.35	84.86
94.94	108.51
101.22	120.73

Numerical data from analysis of carbon columns are presented in tables A-3, and graphs of Kjeldahl nitrogen data are presented in figures 7 through 9. Although Kjeldahl nitrogen data are somewhat scattered, general trends are clear as illustrated by the three figures. The amount of nitrogen detected was partially dependent upon fineness of grinding of carbon samples prior to Kjedahl analysis, presumably because samples were not oxidized during Kjeldahl analysis to the extent that clear solutions were obtained.

TABLE 3. - Organics extracted by CCl_4 and organic nitrogen profiles

Sample location from bottom of column	Organics, pct	Organic nitrogen, ppm	
		Before CCl_4 extraction	After CCl_4 extraction
COLUMN 1			
0 - 2	0.013	0	0
12 - 14	.021	14	6
26 - 28	.036	36	24
38 - 40	.174	121	88
40 - 42	.385	113	ND
42 - 44	.616	188	ND
44 - 46	1.025	239	ND
46 - 48	2.74	276	ND
48 - 50	12.03	438	327
50 - 52	16.04	343	383
COLUMN 2			
0 - 2	.01834	0	ND
16 - 18	.0169	0	ND
34 - 36	.0281	14	ND
50 - 52	.0675	93	ND
54 - 56	.091	128	78
58 - 60	.195	128	90
60 - 62	.385	148	104
62 - 64	.479	136	97
64 - 66	.854	161	96
66 - 68	4.828	228	ND
68 - 68-3/4	12.06	400	ND
COLUMN 3			
0 - 2	.00604	0	9
10 - 12	.00636	4	38
22 - 24	.01086	34	30
42 - 44	.1057	124	97
44 - 46	.209	102	69
46 - 48	.285	128	73
48 - 50	.455	132	94
50 - 52	.731	151	99
52 - 54	2.81	281	210
54 - 55-3/4	11.55	437	332

ND Not determined.

APPENDIX B.--SAMPLE CALCULATIONS

Sample B-1. - Determination of grams organics and grams organics per kilogram CCl_4 from IR absorption peak height

The equations used to determine the grams organics and grams organics per kilogram CCl_4 from IR absorption are as follows:

$$\text{percent transmission} = 100 - \text{peak height}; \quad (1)$$

$$\text{grams organics per kilogram } CCl_4 = 0.7015070 - 0.4704737T + 0.0599882T^2, \quad (2)$$

where T = logarithm percent transmission;

$$\text{Grams organics} = (\text{g org/kg } CCl_4)\ (\text{g } CCl_4)\ (1\ \text{kg}/1{,}000\ \text{g}). \quad (3)$$

The sample data used are as follows:

$$\text{peak height} = 65,$$

$$\text{grams } CCl_4 = 127.19.$$

Using equations 1 through 3 and the sample data, we calculate

$$\text{percent transmission} = 100 - 65,$$

$$= 35;$$

$$T = \text{logarithm percent transmission},$$

$$= \log 35,$$

$$= 1.5441;$$

$$\text{grams organics per kilogram } CCl_4 = 0.7015070 - 0.4704737T + 0.0599882T^2,$$

$$= 0.7015070 - 0.4704737(1.5441) + 0.0599882(1.5441)^2,$$

$$= 0.1181;$$

$$\text{grams organics} = (\text{g org/kg } CCl_4)\ (\text{g } CCl_4)\ (1\ \text{kg}/1{,}000\ \text{g}),$$

$$= (0.1181)\ (127.19)\ (0.001),$$

$$= 0.015.$$

Sample B-2. - A comparison between the IR-determined (corrected) and weighed organics content

To calculate the IR-determined (corrected) and weighed organics content, we use equations 1 through 3 from sample B-1 along with the following equations:

IR-determined organics, ppm × correction factor
= corrected organics contents; (4)

IR-determined extracted organics, ppm = (g org/g liquor) (10^6); (5)

corrected organics content, ppm = extracted organics × correction factor; (6)

weighed organics content (added organic), ppm = (g org added/g liquor) (10^6)
+ soluble organic. (7)

The sample data used are as follows:

correction factor = 0.8534072, (For calculation purposes, the unrounded correction factor is used).

peak height = 80,

weight of liquor extracted = 535.4 g,

weight of CCl_4 used = 120.66 g,

soluble organic in standard liquor sample = 4.01 ppm,

organic added to standard liquor sample = 0.0181 g.

Using equations 1 through 7 and the sample data, we calculate

percent transmission = 100 - 80,

= 20;

T = log pct transmission,

= log 20

= 1.3010;

grams organics per kilogram CCl_4 = 0.1909;

grams organics = 0.0230;

IR-determined extracted organics, ppm = (0.0230 g/535.4 g) (10^6),

= 43.0;

corrected organics content, ppm = 43.0 × 0.8534072,

= 36.7;

weighed organics content (added organic), ppm = (0.0181 g/535.4 g) (10^6)
+ 4.01 ppm,

= 37.8.

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