DEVELOPMENT OF CONTINUOUS SOLVENT EXTRACTION PROCESSES FOR COAL DERIVED CARBON PRODUCTS

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Principal Investigator:
Elliot B. Kennel

Co-Investigators:
Dady B. Dadyburjor, Liviu Magean, J. Joshua Maybury, Alfred H. Stiller, Robert C. Svensson, John W. Zondlo

West Virginia University
Department of Chemical Engineering
College of Engineering and Mineral Resources
PO Box 6102
Morgantown WV 26506

Subcontractors:
GrafTech International
12900 Snow Road
Parma, OH 44130

Koppers Inc.
1005 William Pitt Way
Pittsburgh, PA 15238

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1.0 Executive Summary

This DOE NETL-sponsored effort seeks to develop continuous processes for producing carbon products from solvent-extracted coal. A key process step is removal of solids from liquefied coal. Three different processes were compared: gravity separation, centrifugation using a decanter-type Sharples Pennwalt centrifuge, and a Spinner-II centrifuge. The data suggest that extracts can be cleaned to as low as 0.5% ash level and probably lower using a combination of these techniques.

2.0 Technical

2.1. Gravity Separation

If desired, an initial separation can be accomplished simply using gravity separation. The solids are slightly heavier than the liquid phase and thus accumulate as a sludge on the bottom of the barrel.

The drum should be heated prior to decanting. The temperature is likely limited by the capability of the double diaphragm pump (typically 65 ^{o}C for polymer body pumps). There are two main reasons for heating. The first reason is to melt and solubilize any polymerized but usable phases that might be present in the sludge. The second reason is to allow suspended particles present in the nominal liquid phase to settle to the bottom. Lower viscosity results in improved separation.

Each drum of un-centrifuged extract may be decanted by holding the pump P1 slightly above the bottom sludge level to the "work drum" D1. The bottom sludge should be retained and consolidated. Table 1 illustrates results from an unfiltered extract drum. Samples were taken from near the top of the drum, in approximately the middle and near the bottom of the sludge layer. Thus the results suggest that ash levels in the upper liquid phase are moderate simply by allowing gravity separation.

Table 1. Results of Gravity Separation; Liquid Phase.

Name	Crucible Mass	Initial Mass	Location	Method	Analysis Date	Moisture	Volatile	Ash
Near top 1	13.433	1.7395	1	htv	10/31/2006 15:50	8.84	73.21	0.79
Near top 2	15.25	1.8701	2	htv	10/31/2006 15:50	8.32	73.71	0.79
Near top 3	15.371	2.1803	3	htv	10/31/2006 15:50	7.7	73.51	1.13
Near middle 1	15.385	2.0541	4	htv	10/31/2006 15:50	8.27	73.94	0.72
Near middle 2	14.041	1.6221	5	htv	10/31/2006 15:50	9.2	72.75	0.81
Near middle 3	13.727	1.6765	6	htv	10/31/2006 15:50	9.21	73.55	0.58
Near bottom 1	15.885	1.5909	7	htv	10/31/2006 15:50	9.4	72.47	0.85
Near bottom 2	13.865	1.9153	8	htv	10/31/2006 15:50	8.36	73.9	0.67
Near bottom 3	13.402	1.8232	9	htv	10/31/2006 15:50	8.82	73.55	0.65

2.2 Centrifugation Using a Sharples Pennwalt Decanter Centrifuge

2.2.1 Basic System Configuration

The system consists of a Sharples Pennwalt P660 Super D-canter centrifuge that is gravity fed via an 8-gallon tank. The feeder system consists of a Gate Valve for flow control to the Pennwalt, a double diaphragm pump to fill the ballast tank, and a holding tank for the Pennwalt output. Another diaphragm pump is used to pump the product from the holding tank back to the 55-gallon drum, which holds product to be centrifuged. The Pennwalt product output can easily be transferred to another drum; i.e., a clean product drum (D2).

The double diaphragm pump has been proven to be suitable for pumping coal slurries. However, this pump is a pulsed device, whereas the manufacturer recommends an approximately constant flow to the centrifuge inlet. For this reason a pressurized ballast tank is used. The flow from the ballast tank is approximately constant.

The centrifuge can also be operated in a recirculating mode, to allow the working fluid to pass through several times in order to achieve very low solids content.

2.2.2 Detailed Description of the System

The medium to be cleaned is pumped from a 55 gallon drum (D1) with the aid of an air driven diaphragm pump (P1) via valves (V1, V6) up to a feed tank (T1) that is located around 2 ft. above the Pennwalt. The elevated location allows the product to be gravity fed into the Pennwalt from T1 via the shut-off valve (V2) and the Gate Valve (Gate), which is mounted on the Pennwalt input pipe with a union. In the Pennwalt the liquid and the solids are separated. The liquid exits the Pennwalt in the gearbox end of the rotor unit via a valve (V3) and runs down to a holding tank (T2) with gravity forces only. The solids (or centrifuge tails) are dumped into a 19" sludge drum (SD) at the pulley end. From T2 the liquid is pumped back to D1 (or D2) with the pump P2.

Valve V5 is used to transfer product from one drum to another without passing T1. This route is of no significance for the separation process itself.

The feed tank can be used as a gravity feeder tank or for pressurized feeding tank. When used as a gravity feeder, the product is continuously pumped into the tank and an overflow valve (V4) is full open. The overflow product is drained back to D1. Thus, the level in the T1 is held constant. Due to the constant level, it is possible to calibrate the flow thru Gate as a function of the number of turns of the control wheel. A calibration table has been made.

When using T1 as a pressure feeder, the pressure is built up by P1 (or by the means of an N_2 cylinder located in the control room, a procedure that probably is redundant). When used as a pressure feeder, the pressure drop dp/dt gives the flow as a function of time.

The feed tank is equipped with one pressure gage with a Startup range of 0-60psig (G1) and one with a range of 0-200psig (G2). G1 is the most important one due to its sensitivity for low-pressure changes. There is a pressure relief valve (PRV) on the gas line that can be used to depressurize T1. There is also an over-pressure valve (OPV).

The power for the compressor is tapped from of two of the three phases in one of the two existing 3 PH 220 VAC. The Space Heater should be connected between two of the remaining phases of the receptacle used for the compressor. There is a possibility (technically correct) to use three different single-phase 208/220-240V loads, each one drawing 20Amps.

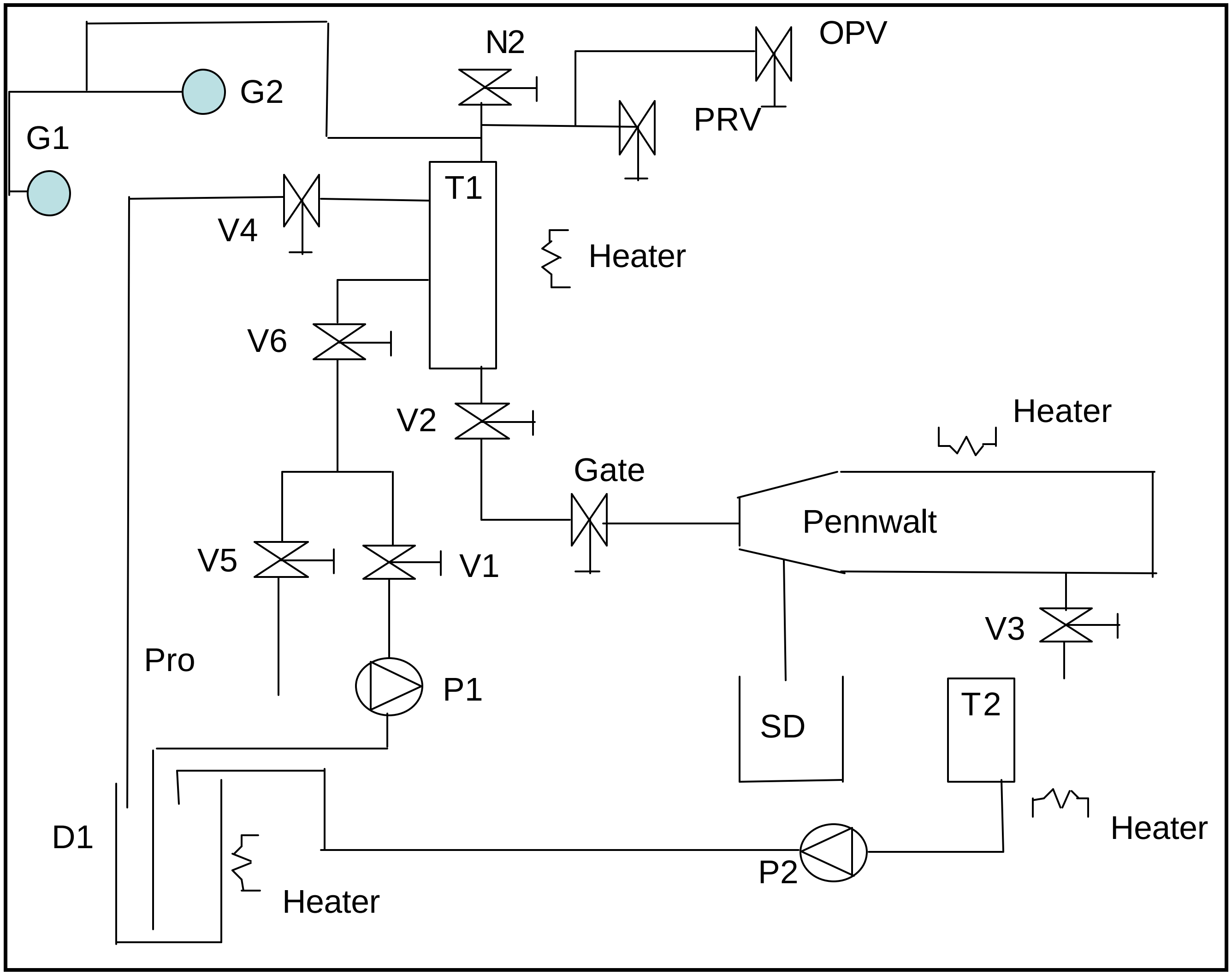

Figure 1. Sharples-Pennwalt Centrifuge Configuration.

Figure 2. Installation of the Sharples Pennwalt Decanter-Type Centrifuge.

Table 2. Elemental Analysis of Kingwood Coal.

Date :	10/25/2006	09:24:52
Method Name :	NCHS	
Method Filename :	102406.mth	

Group No : 1	Element %			
Sample Name	Nitrogen%	Carbon%	Hydrogen%	Sulphur%
KINGWOOD COAL	1.736782312	79.2086792	5.318805695	1.379725337
KINGWOOD COAL	1.720533609	79.49386597	5.248065948	1.226955414
KINGWOOD COAL	1.682213068	78.1993103	5.129944801	1.153113961

3 Sample(s) in Group No : 1

Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	1.71317633	0.0280187	1.6355	0.0008
Carbon%	78.96728516	0.6801999	0.8614	0.4627
Hydrogen%	5.232272148	0.09541589	1.8236	0.0091
Sulphur%	1.253264904	0.1155739	9.2218	0.0134

Table 3. Elemental Analysis of Spinner-II Model 60 Solids, 10/18/2006.

Group No : 3	Element %			
Sample Name	Nitrogen%	Carbon%	Hydrogen%	Sulphur%
CENT SOLIDS 10/18	0.958836734	86.70681	5.603644848	0.577280521
CENT SOLIDS 10/18	0.922740221	86.36791229	5.682459831	0.586459339
CENT SOLIDS 10/18	0.967725694	86.23344421	5.629908085	0.570852578

3 Sample(s) in Group No : 3				
Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	0.94976755	0.02382458	2.5085	0.0006
Carbon%	86.4360555	0.2439291	0.2822	0.0595
Hydrogen%	5.638670921	0.04013155	0.7117	0.0016
Sulphur%	0.578197479	0.007843683	1.3566	0.0001

Table 4. Elemental Analysis of Spinner-II Model 60 Solids 10/19/2006.

Group No : 4	Element %			
Sample Name	Nitrogen%	Carbon%	Hydrogen%	Sulphur%
CENT SOLIDS 10/19	0.950949252	86.75003815	5.624344826	0.545578778
CENT SOLIDS 10/19	0.948956609	86.09523773	5.568933487	0.549670339
CENT SOLIDS 10/19	0.948690951	86.20910645	5.604275227	0.554657817

3 Sample(s) in Group No : 4				
Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	0.94953227	0.00123431	0.13	0
Carbon%	86.35146077	0.3498421	0.4051	0.1224
Hydrogen%	5.599184513	0.02805424	0.501	0.0008
Sulphur%	0.549968978	0.004546881	0.8268	0

Table 5. Elemental Analysis of Spinner-II Model 60 Solids, 10/20/2006.

Group No : 5	Element %			
Sample Name	Nitrogen%	Carbon%	Hydrogen%	Sulphur%
CENT SOLIDS RUN1 10/20	0.957164645	87.01338196	5.651396275	0.526128769
CENT SOLIDS RUN1 10/20	1.030314565	87.51313019	5.565959454	0.539865851
CENT SOLIDS RUN1 10/20	0.946367443	86.56817627	5.614240646	0.541499853

3 Sample(s) in Group No : 5				
Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	0.977948884	0.04567022	4.67	0.0021
Carbon%	87.03156281	0.4727392	0.5432	0.2235
Hydrogen%	5.610532125	0.04283897	0.7635	0.0018

Sulphur%	0.535831491	0.008442429	1.5756	0.0001

Table 6. Elemental Analysis of Spinner-II Model 60 Solids from 10/20 (2nd Trial).

Group No : 6	Element %			
Sample Name	Nitrogen%	Carbon%	Hydrogen%	Sulphur%
CENT SOLIDS RUN2 10/20	0.868736982	85.89995575	5.451105118	0.544663191
CENT SOLIDS RUN2 10/20	0.916819751	86.00952148	5.542014599	0.577025473
CENT SOLIDS RUN2 10/20	0.927151978	85.73941803	5.58523941	0.569873214

3 Sample(s) in Group No : 6				
Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	0.904236237	0.03117429	3.4476	0.001
Carbon%	85.88296509	0.135851	0.1582	0.0185
Hydrogen%	5.526119709	0.06846523	1.2389	0.0047
Sulphur%	0.563853959	0.01700008	3.015	0.0003

Table 7. Elemental Analysis of Spinner-II Model 60 Centrate 10/20/2006.

Group No : 2	Element %			
Sample Name	Nitrogen%	Carbon%	Hydrogen%	Sulphur%
CENT LIQUID 10/20	0.718712926	90.23823547	6.331259251	0.300561547
CENT LIQUID 10/20	0.790115714	90.46909332	6.26751852	0.266353607
CENT LIQUID 10/20	0.785581887	90.69264221	6.054850101	0.238526046

3 Sample(s) in Group No : 2				
Component Name	Average	Std. Dev.	% Rel. S. D.	Variance
Nitrogen%	0.764803509	0.03997993	5.2275	0.0016
Carbon%	90.466657	0.2272132	0.2512	0.0516
Hydrogen%	6.217875957	0.144737	2.3278	0.0209
Sulphur%	0.2684804	0.03107239	11.5734	0.001

2.2.3 Start-up Procedure

The decanter centrifuge cannot be run at full rpm unless it is warmed up to above room temperature. If it is attempted to run the centrifuge at full rpm at lower temperatures with viscous fluids (e.g., coal extract), the motor controller shuts it down due to the high viscosity of the coal-derived working fluids and correspondingly high torque. Hence, before starting, the centrifuge must be heated using a the space heater and drum band heater. Diesel oil @ approx. 50 degrees Celsius can be used as a substitute working fluid as the system is warmed up. The opening of Gate should be around 2 turns when diesel is used. Some of the diesel is deposited in the sludge drum and the solids pathway is also cleaned.

The centrifuge must first be brought up to operating speed; or around 5,600 rpm. The manufacturer's limit is 6,000 rpm, which should not be exceeded.

The diesel oil is pumped up into T1 with V2 closed and V4 full open. The Gate is set to approximately 2-3 turns. When diesel oil is observed exiting the V4 hose, V2 should be opened. Diesel is then gravity fed into the centrifuge. The system is kept running until the rotor cover of the Pennwalt is about 50 ^{o}C. The Main Bearing temperature is monitored, and when that is around 30 deg. Celsius, the centrifuge rotational speed is increased to 5,600 rpm with the up-down keys at the controller. To shut down the diesel procedure, P1 is shut off. When nothing is coming out from the return hose, V2 can be closed.

Because the facility containing the centrifuge has high air throughput, it can become cold if the weather outdoors is cold. In this situation, the room should be brought up to 70 ^{o}F using space heaters and held for at least one hour prior to operating the centrifuge.

A variant of this procedure would be to use NMP solvent in place of diesel fuel. NMP has the advantage of having a higher flash point (95 ^{o}C versus 62 ^{o}C), as well as a less pungent odor. However, if NMP is used, it is necessary to follow it with a few gallons of coal tar distillate to ensure that NMP does not mix with the product, because the heteroatoms of NMP might cross-link atoms in the product, resulting in a more isotropic product.

At this point the system is ready to operate on the two phase coal extract slurry.

2.2.4 Normal Operating Procedure.

Pump P1 is moved to the product feed drum. The overflow and return hoses from the diesel drum are moved to the product drum. P1 is started at full capacity with V4 fully open. When product is observed from the return hose, the pump speed is reduced such that the flow is even. V2 is then opened. The Gate is turned to approximately 2 turns (the Gate is turned past the desired value and then backed down to the desired value). During the run it is highly advisable to monitor the flow from the hoses.

A mass balance is obtained by weighing the un-centrifuged extract prior to centrifugation, then weighing the mass of the centrifuged product as well as the mass of the centrifuge tails. A proximate analysis should be performed on all three material streams to determine the ash levels.

The goal for this effort is to produce low ash levels in products such as binder pitch, anode coke and needle coke.

$$A_{SPEC} > A_{Koppers}V_{Koppers} + A_{SP}V_{SP} \quad ,$$

where A_{SPEC} is the customer-specified ash limit, $A_{Koppers}$ is the amount of ash contained in Koppers control coal tar binder pitch, $V_{Koppers}$ is the volume fraction of Koppers coal tar pitch, A_{SP} is the ash content in the synthetic pitch, and V_{SP} is the volume fraction of synthetic pitch.

As a rule of thumb, 0.5% ash content or lower is desired. Ash content is characterized via proximate analysis. That is, ash content actually refers to the amount of material present after complete oxidation, divided by the dry mass initially present.

Higher values of ash content might be tolerated if the Koppers control pitch has an even lower quantity of ash, as it normally does. For example, in the present project it is intended that synthetic binder pitch be blended in a 20/80 ratio with Koppers coal tar binder pitch. Hence if Koppers coal tar binder pitch has a value of 0.2% ash, in order to achieve the proper specifications,

$$0.005 > 0.002*0.80 + A_{SP}* 0.20 \quad ,$$

$$A_{SP} < \sim 1.7\% \quad .$$

The ash level in the un-centrifuged extract is determined by the yield of synpitch after distillation, estimated to be 30%. Hence

$$A_{ext} < A_{SP}*0.30$$

or

$$A_{ext} < 0.51\ \% \quad .$$

This represents the upper limit of the permissible ash levels, and depends upon the Koppers control material to be cleaner than the synthetic pitch. If on the other hand, the synthetic pitch were to achieve the same level of ash as the control, then the ash level of the un-separated extract would need to be 0.067%.

In order to judge the effectiveness of separation, the ash content of the extract must be compared to the ash content in the tails. The ability of the centrifuge to concentrate the ash may be identified as the ratio between the ash content in the extract divided by the ash content in the tails; that is,

$$\eta = A_{tails} / A_{ext} \, ,$$

where η is the ratio of the ash content in the extract (liquid output from the centrifuge or centrate) A_{ext} and the ash content in the tails, A_{tails}.

In this case it is desirable to have as large a value of η as possible. Based on literature values for centrifugation, the tails can be 85% liquid, and 15% solid (though this has not been achieved experimentally in the present effort). The tails are assumed to consist partly of mineral matter and partly of fixed carbon, in approximately equal amounts. Thus as a goal,

$$\eta > (0.5 * 0.85) / 0.0051$$

$$\eta > 83 .$$

This may be difficult to achieve in practice, however. Pennwalt India (the successor to Sharples Pennwalt) advises that concentration ratios greater than about 10 often require the use of a second centrifuge in series to accomplish.

If lower centrifuge concentration ratios are achieved, the practical interpretation of this is that the amount of product lost in the tails is increased. For example, tests with a Spinner II centrifuge resulted in a measured ash concentration of about 15% in the tails, and 0.0038 in the centrate. This results in $\eta = 39$. Assuming that the coal contains 7.0 % ash, and that the ratio of distillate mass to coal mass is 3:1, then the extract should contain 1.75% ash. By equating the ash in the tails to the ash in the original un-centrifuged extract,

$$m_{tails} A_{tails} = m_{ext} A_{ext} \quad .$$

Thus,

$$m_{tails} = \frac{m_{ext} A_{ext}}{A_{tails}} \quad ,$$

or

$$m_{tails} = 11.7\% \, m_{ext}$$

The amount of product "lost" (that is, the amount of product identical to the centrate that winds up in the tails stream rather than the nominal centrate stream) depends upon how much non-ash, solid material exists in the extract. That is, it is assumed that there is a certain amount of solid carbon that is not easily separable from the ash, and thus must be included in the tails stream rather than the centrate stream.

For this case, it is assumed that the mass of fixed carbon is approximately equal to the mass of ash. Thus, the amount of liquid contained in the tails stream is approximately

$$m_{tailsliquid} = \frac{m_{ext} A_{ext}}{A_{tails}} \left(1 - 2A_{ext}\right) \quad .$$

Or, for the specific example described above,

$$m_{tailsliquid} = 8.2\% \, m_{ext} \quad .$$

Thus for a 450 lb sample, approximately 37 pounds of liquid will be incorporated in the tails.

2.2.5 Additional Data Needed

A mass balance similar to the analysis accomplished for the Spinner II Model 60 needs to be performed for the Sharples-Pennwalt centrifuge to determine the ash removal rate, ash concentration in the tails, and ash concentration in the centrate (see Section 2.3).

It is believed that the "pond depth" setting in the unit should be adjusted. The pond depth refers to the depth of the annual volume of liquid that is created in the rotating centrifuge. Currently the pond depth is set at the minimum, which should result in the cleanest possible centrate. However, it is hypothesized that the pond depth should be maximized in order to result in the maximum possible solids concentration in the tails. Then the centrate can be run through the system in multiple passes in order to achieve lower ash levels. Alternatively, a second polishing centrifuge can be used to reduce ash levels further. This protocol will likely result in clean centrate with a maximum concentration of ash in the tails, thus minimizing product losses.

2.3 Centrifugation Using a Spinner II Polishing Centrifuge

2.3.1 Basic System Configuration

A Spinner-II centrifuge was selected as a means for further reducing the ash content in coal tar slurries. The Spinner-II was originally designed for cleaning engine oil in diesel engines, but has been adapted for a number of applications. Tests were first carried out in a Spinner II Model 60 to demonstrate the suitability of this type of centrifuge for the particular materials encountered in solvent extraction of coal. The Spinner II Model 60 unit is small and inexpensive (under $1000), but according to the manufacturer is able to remove submicron particles from liquid streams.

Referring to Figure 3 below, the basic process is as follows:

1) Dirty oil enters the separation chamber under normal pressure, flowing up through a hollow spindle.

2) Oil passes through a spinning rotor where 2000 g centrifugal force separates contaminants from the oil.

3) Contaminants accumulate on the rotor surface as a solid cake (see Figure 4).

4) Clean oil exits through opposing, twin nozzles that power the centrifuge up to 4,000 rpm.

5) Clean oil returns to the sump/reservoir from the level control base.

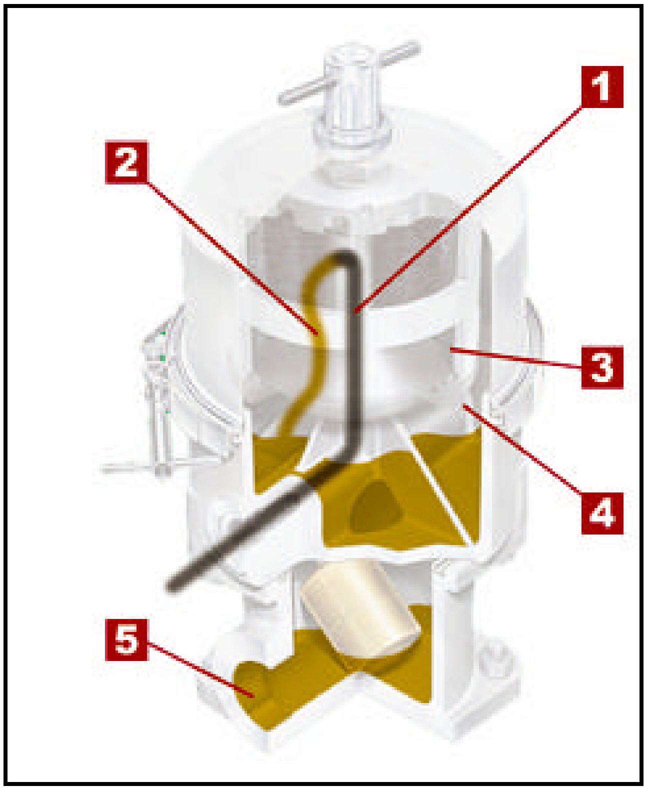

Figure 3. Spinner II Centrifuge Conceptual Design.

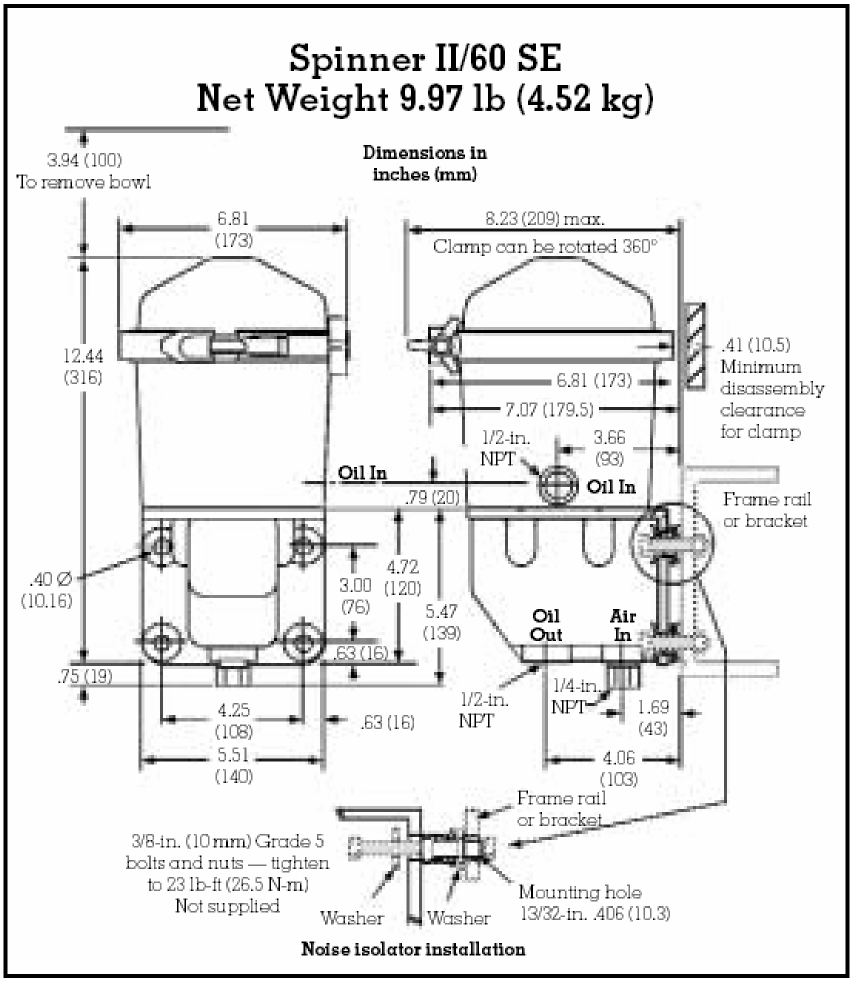

Figure 4. Details of the Spinner-II Model 60.

Figures 5 and 6 show the Spinner-II Model 60 setup. Figure 6 is the manufacturer's spec for the pressure flow relationship.

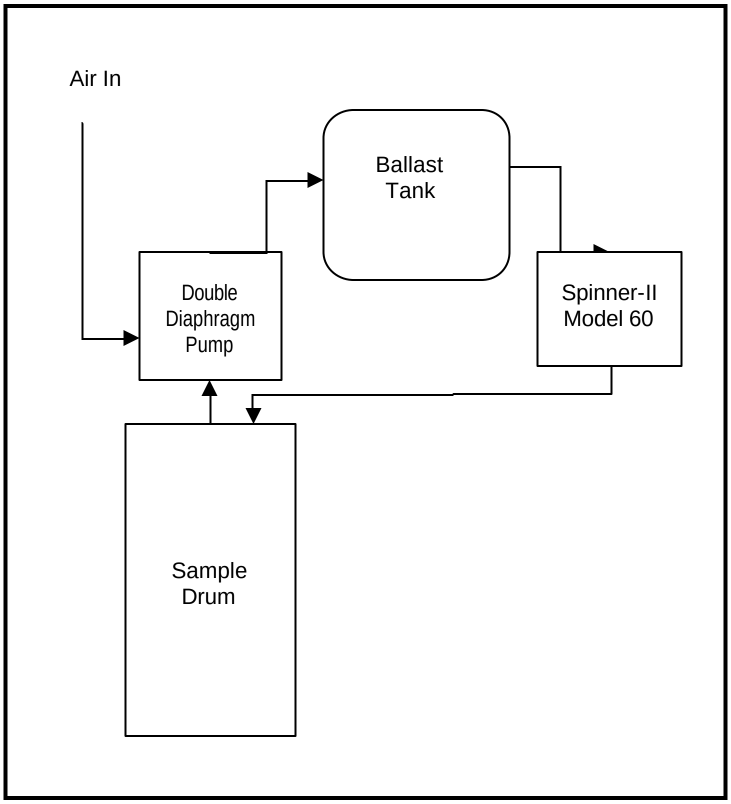

Figure 5. Spinner II Model 60 System Schematic.

Figure 6. Spinner II Model 60 Centrifuge Experimental Setup.

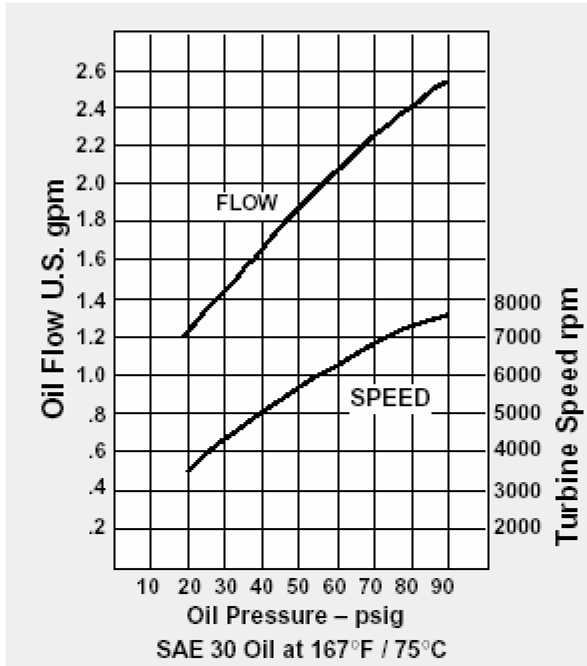

Figure 7. Flow-Pressure Relationship for Spinner II Model 60.

Tables 8-29 contain proximate analyses of the centrate as well as the tails from the Spinner II Model 60 unit. The results are summarized in Table 30 and depicted graphically in Figures 8 and 9. The disassembled unit is shown in Figure 10, showing the centrifuge tails collected in the form of a semi-solid cake.

The results show that the extract is progressively cleaned as it circulates. A larger unit with higher throughput capability would presumably achieve comparable or better results in less time. Thus a larger Model 600 unit was purchased (see Section 2.3.1).

Table 8. Proximate Analysis of Spinner-II Model 60Tails 11-10-2006.

Sample Name	Sample Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 11-10-06 1420 1	2.0218	13	11/16/2006 16:13	3.85	59.00	10.94	26.21
Tails 11-10-06 1420 2	2.0188	14	11/16/2006 16:13	3.89	58.43	11.27	26.41
Tails 11-10-06 1420 3	2.1359	15	11/16/2006 16:13	3.87	59.10	10.78	26.25
Tails 11-10-06 1420 avg	2.0588		11/16/2006 16:13	3.87	58.84	11.00	26.29

Table 9. Proximate Analysis of Spinner-II Model 60 Centrate, 11-10-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 11-10-06 1420 1	1.4831	10	11/16/2006 16:13	7.80	73.19	0.83	18.18
Extract 11-10-06 1420 2	1.4698	11	11/16/2006 16:13	7.35	73.71	0.94	18.00
Extract 11-10-06 1420 3	1.3970	12	11/16/2006 16:13	7.63	73.78	0.95	17.64
Extract 11-10-06 1420 avg	1.4500		11/16/2006 16:13	7.59	73.56	0.91	17.94

Table 10. Proximate Analysis of Spinner-II Model 60 Tails, 2nd Sample, 11-10-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 11-10-06 1630 1	1.8059	7	11/16/2006 16:13	4.28	57.92	10.08	27.72
Tails 11-10-06 1630 2	2.2322	8	11/16/2006 16:13	4.07	58.09	10.17	27.67
Tails 11-10-06 1630 3	2.3826	9	11/16/2006 16:13	3.28	59.46	9.97	27.29
Tails 11-10-06 1630 avg	2.1402		11/16/2006 16:13	3.88	58.49	10.07	27.56

Table 11. Proximate Analysis of Spinner-II Model 60 Centrate, 2nd Sample, 11-10-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 11-10-06 1630 1	1.3882	4	11/16/2006 16:13	7.58	73.46	0.74	18.22
Extract 11-10-06 1630 2	1.5460	5	11/16/2006 16:13	7.34	74.18	0.54	17.94
Extract 11-10-06 1630 3	1.5645	6	11/16/2006 16:13	7.04	74.00	0.70	18.26
Extract 11-10-06 1630 avg	1.4996		11/16/2006 16:13	7.32	73.88	0.66	18.14
Extract 11-10-06 1630 retest 1	1.3487	3	1/5/2007 15:37	7.79	74.82	0.33	17.06
Extract 11-10-06 1630 retest 2	1.4794	4	1/5/2007 15:37	6.92	75.48	0.53	17.07
Extract 11-10-06 1630 retest avg	1.4141		1/5/2007 15:37	7.36	75.15	**0.43**	17.07

Table 12. Proximate Analysis of Spinner-II Model 60 Tails, 11-16-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 11-16-06 1055 1	1.9409	6	11/21/2006 16:29	4.01	67.34	5.43	23.22
Tails 11-16-06 1055 2	1.7786	7	11/21/2006 16:29	4.24	66.08	5.67	24.01
Tails 11-16-06 1055 avg	1.8598		11/21/2006 16:29	4.13	66.71	5.55	23.62

Table 13. Proximate Analysis of Spinner-II Model 60 Centrate, 11-16-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 11-16-06 1055 1	1.2545	4	11/21/2006 16:29	7.38	73.32	0.69	18.61
Extract 11-16-06 1055 2	1.7792	5	11/21/2006 16:29	6.00	74.58	0.82	18.60
Extract 11-16-06 1055 avg	1.5169		11/21/2006 16:29	6.69	73.95	0.76	18.61

Table 14. Proximate Analysis of Spinner-II Model 60 Tails, 2nd Sample, 11-16-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 11-16-06 1240 1	1.7738	10	11/21/2006 16:29	5.21	61.55	6.66	26.58
Tails 11-16-06 1240 2	2.0249	11	11/21/2006 16:29	4.46	62.28	6.84	26.42
Tails 11-16-06 1240 avg	1.8994		11/21/2006 16:29	4.84	61.92	6.75	26.50

Table 15. Proximate Analysis of Spinner-II Model 60 Centrate, 2nd Sample, 11-16-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 11-16-06 1240 1	1.7906	8	11/21/2006 16:29	6.27	73.87	0.72	19.14
Extract 11-16-06 1240 2	1.4951	9	11/21/2006 16:29	6.93	72.99	0.74	19.34
Extract 11-16-06 1240 avg	1.6429		11/21/2006 16:29	6.60	73.43	0.73	19.24

Table 16. Proximate Analysis of Spinner-II Model 60 Tails, 11-17-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 11-17-06 1430 1	2.3119	5	11/27/2006 17:30	3.92	61.84	6.30	27.94
Tails 11-17-06 1430 2	2.6673	6	11/27/2006 17:30	4.01	61.62	6.12	28.25
Tails 11-17-06 1430 avg	2.4896		11/27/2006 17:30	3.97	61.73	6.21	28.10

Table 17. Proximate Analysis of Spinner-II Model 60 Centrate, 11-17-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 11-17-06 1430 1	1.7418	3	11/27/2006 17:30	6.31	72.53	0.61	20.55
Extract 11-17-06 1430 2	1.7537	4	11/27/2006 17:30	6.21	73.25	0.59	19.95
Extract 11-17-06 1430 avg	1.7478		11/27/2006 17:30	6.26	72.89	0.60	20.25

Table 18. Proximate Analysis of Spinner-II Model 60 Tails, 12-01-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 12-01-06 1430 1	2.3297	3	12/5/2006 16:05	3.79	73.45	3.39	19.37
Tails 12-01-06 1430 2	2.1248	4	12/5/2006 16:05	5.07	69.49	3.99	21.45
Tails 12-01-06 1430 avg	2.2273		12/5/2006 16:05	4.43	71.47	3.69	20.41

Table 19. Proximate Analysis of Spinner-II Model 60 Centrate, 12-01-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 12-01-06 1430 1	1.4879	5	12/5/2006 16:05	8.04	74.86	0.52	16.58
Extract 12-01-06 1430 2	1.7607	6	12/5/2006 16:05	7.58	75.54	0.51	16.37
Extract 12-01-06 1430 avg	1.6243		12/5/2006 16:05	7.81	75.20	0.52	16.48

Table 20. Proximate Analysis of Spinner-II Model 60 Tails, 12-07-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 12-07-06 1045 1	1.1873	6		7.56	65.47	4.03	22.94
Tails 12-07-06 1045 2	1.8425	7		4.90	68.31	4.06	22.73
Tails 12-07-06 1045 avg	1.5149			6.23	66.89	4.05	22.84

Table 21. Proximate Analysis of Spinner-II Model 60 Centrate, 12-07-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 12-07-06 1045 1	1.3048	8		7.90	75.07	0.52	16.51
Extract 12-07-06 1045 2	1.5219	9		7.60	75.60	0.45	16.35
Extract 12-07-06 1045 avg	1.4134			7.75	75.34	0.49	16.43

Table 22. Proximate Analysis of Spinner-II Model 60 Tails, 2nd Sample, 12-07-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Tails 12-07-06 1355 1	1.4715	1		5.58	64.03	4.75	25.64
Tails 12-07-06 1355 2	1.3722	2		6.89	62.66	4.81	25.64
Tails 12-07-06 1355 3	1.6735	3		5.33	64.76	4.60	25.31
Tails 12-07-06 1355 avg	1.5057			5.93	63.82	4.72	25.53

Table 23. Proximate Analysis of Spinner-II Model 60 Centrate, 2nd Sample, 12-07-2006.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Extract 12-07-06 1355 1	1.3353	4		7.57	75.58	0.39	16.46
Extract 12-07-06 1355 2	1.2291	5		7.54	75.71	0.37	16.38
Extract 12-07-06 1355 avg	1.2822			7.56	75.65	0.38	16.42

Table 24. Proximate Analysis of Spinner-II Model 60 Tails, 1-02-2007.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Spinner II tails 1/02/07 1200 1	1.7900	7	1/5/2007 15:37	4.25	77.97	1.86	15.92
Spinner II tails 1/02/07 1200 2	1.9060	8	1/5/2007 15:37	4.68	77.43	1.82	16.07
Tails 12-07-06 1045 avg	1.8480		1/5/2007 15:37	4.47	77.70	1.84	16.00

Table 25. Proximate Analysis of Spinner-II Model 60 Centrate, 1-02-2007.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Spinner II extract 1/02/07 1200 1	1.4323	5	1/5/2007 15:37	7.01	75.82	0.47	16.70
Spinner II extract 1/02/07 1200 2	1.7895	6	1/5/2007 15:37	6.27	76.81	0.35	16.57
Extract 12-07-06 1355 avg	1.6109		1/5/2007 15:37	6.64	76.32	0.41	16.64

Table 26. Proximate Analysis of Spinner-II Model 60 Tails, 1-03-2007.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Spinner II tails 1/03/07 1315 2	1.9665	2	1/8/2007 15:38	4.70	66.69	3.80	24.81
Spinner II tails 1/03/07 1315 3	1.7107	3	1/8/2007 15:38	4.53	66.90	3.76	24.81
Tails 12-07-06 1045 avg	1.8386		1/8/2007 15:38	4.62	66.80	3.78	24.81

Table 27. Proximate Analysis of Spinner-II Model 60 Centrate, 1-03-2007.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Spinner II extract 1/03/07 1315 1	1.5961	9	1/5/2007 15:37	7.22	75.95	0.39	16.44
Spinner II extract 1/03/07 1315 2	1.6813	10	1/5/2007 15:37	7.09	75.98	0.39	16.54
Extract 1/03/2007 1315 avg	1.6387		1/5/2007 15:37	7.16	75.97	0.39	16.49

Table 28. Proximate Analysis of Spinner-II Model 60 Tails, 1-04-2007.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Spinner II tails 1/04/07 1305 1	2.6009	15	1/5/2007 15:37	3.34	66.74	1.12	28.80
Spinner II tails 1/04/07 1305 2	2.0377	16	1/5/2007 15:37	3.40	67.04	0.84	28.72
Tails 1/04/2007 1305 avg	2.3193		1/5/2007 15:37	3.37	66.89	0.98	28.76

Table 29. Proximate Analysis of Spinner-II Model 60 Centrate, 1-04-2007.

Sample Name	Initial Mass	Location	Analysis Date	Moisture	Volatile	Ash	Free Carbon
Spinner II extract 1/04/07 1305 1	1.4920	4	1/8/2007 15:38	7.67	73.92	0.41	18.00
Spinner II extract 1/04/07 1305 2	1.5043	5	1/8/2007 15:38	7.42	73.77	0.41	18.40
Extract 1/04/07 1305 avg	1.4982		1/8/2007 15:38	7.55	73.85	0.41	18.20

Table 30. Proximate Analysis Summary.

Time (hrs)	0.00	0.25	1.25	2.75	3.75	5.25	6.25	7.25	9.25	11.25	15.25	19.25
Tails Ash Level		11.00	10.07	5.55	6.75	6.21	3.69	4.05	4.72	1.84	3.78	0.98
Extract Ash Level		0.91	0.66	0.76	0.73	0.60	0.52	0.49	0.38	0.41	0.39	0.41
Minutes / pass	15	60	90	60	90	60	60	120	120	240	240	
Time (hrs)	0.00	0.25	1.25	2.75	3.75	5.25	6.25	7.25	9.25	11.25	15.25	19.25

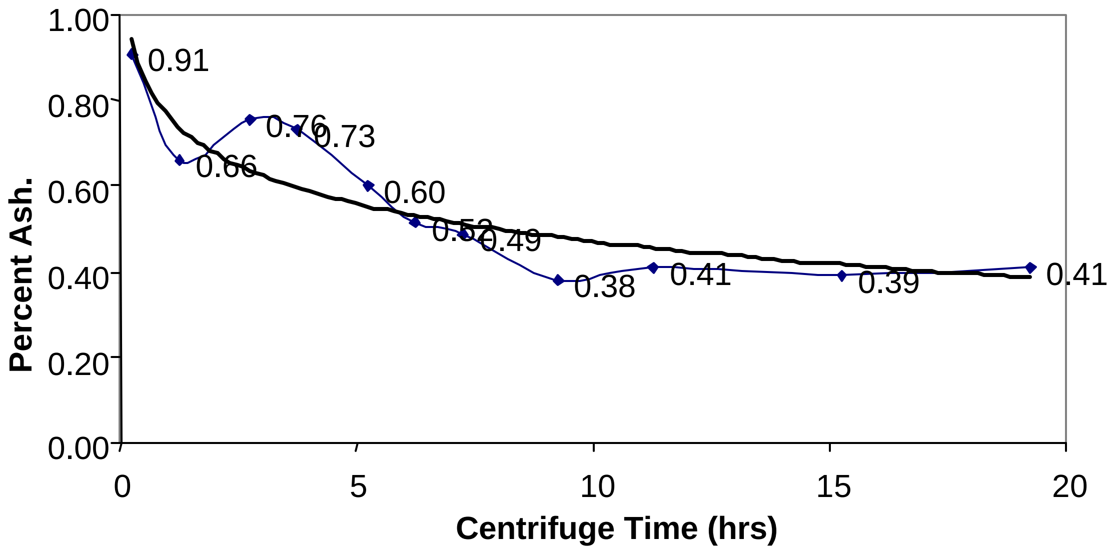

Figure 8. Ash Content of the Spinner II Centrate as a Function of Run Time.

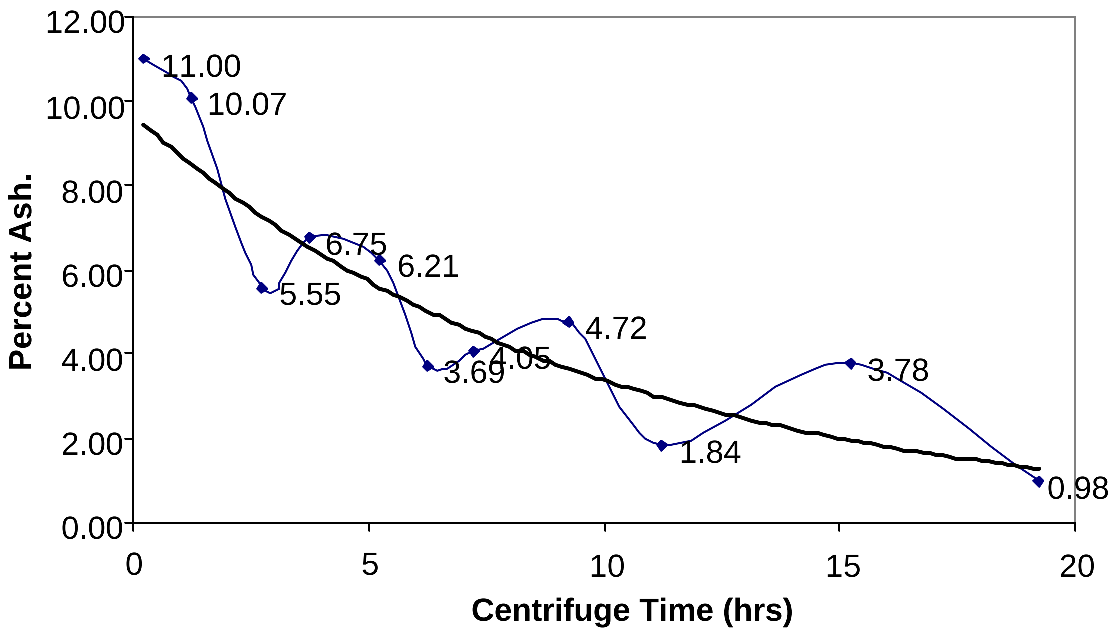

Figure 9. Ash Content of the Spinner-II Model 60 Tails as a Function of Run Time.

Figure 10. The Cover Removed from the Spinner II Model 60, showing the solid "Cake."

There is significant scatter in the data. This may be due to inhomogenous sampling, or possibly unintentional separation during the handling process. There is also a possibility that mass measurements in the LECO analyzer may contain variability due to excess vibration (i.e., a poorly functioning cooling fan). Accordingly, the unit was serviced in late December 2006. The LECO unit is shown in Figure 11 below.

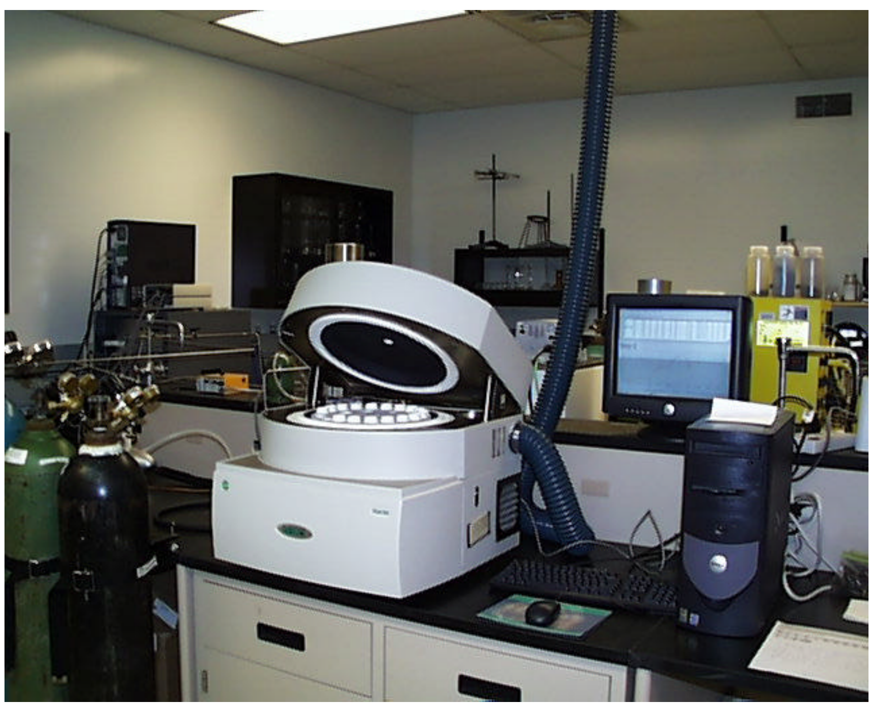

Figure 11. LECO Proximate Analyzer.

2.3.2 Spinner II Model 600

Based on the results of the tests with the Spinner II Model 60, a model 600 unit was ordered. The main difference is that the Model 600 unit can contain 6.0 liters of solids, versus 0.2 liters for the Model 60. This is believed to be suitable for processing a 55 gallon drum of extract. The unit had to be backordered, however, and results were not available in time to be included in this report. The rotor and housing are shown below in Figure 12.

Figure 12. Spinner-II Model 600 Centrifuge Assembly.

3.0 References

None.