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Fluor MPR-77-10

COAL LIQUEFACTION PILOT PLANT

CRESAP, WEST VIRGINIA

MONTHLY TECHNICAL PROGRESS REPORT

OCTOBER, 1977

Fluor Engineers and Constructors, Inc.

3333 Michelson Drive

Irvine, California 92730

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PREPARED FOR THE UNITED STATES

DEPARTMENT OF ENERGY

Under Contract No. EX-76-C-01-1517

NOTIFICATION OF TRIPS

Name	Duration	Destination	Purpose
G. O. Fredrickson	10/6-10/21	Cresap, West Va.	ERDA Meeting & LCDC Support
F. U. Leonard	10/10-10/28	Cresap, West Va.	ERDA Meeting
L. Gasendo	10/17-10/23	Cresap, West Va.	Plant Startup

I. OPERATIONS

A. Summary

Coal feed was maintained to the extraction end of the pilot plant over a period of twenty hours. Approximately four tons of coal were processed at 9:1 solvent-to-coal ratio. Very limited data show the performance of the Solids Separation and Low Temperature Carbonization sections to have been satisfactory. Extraction depth, less than 50 percent, was limited by low temperatures in the extractor. Line blockages in the Solvent Recovery Section forced a halt to the run when the extract granulation system did not work, and all other means for extract removal became plugged.

The preferred process conditions for the Solids Separation and Solvent Recovery sections will be developed during next month's operations of the front end of the pilot plant.

An earlier brief attempt to process coal was interrupted by a major leak at the inlet to the slurry preheater. During the subsequent emergency shutdown, the preheater coil plugged with coke. The middle section of the preheater was replaced with the spare coil. Other operating delays resulted from two failures of the extractor agitator shaft seals prior to the introduction of coal.

Most of the leaky Conval block valves in the Hydrotreating Section have been identified; repairs will be completed next month. Loose, gritty material will be flushed from the system with condensate. Following a reassessment of the relief valve situation with the Vendor, pressure testing will proceed at 2000 psig.

The International Chemical Workers Union has filed a complaint with the National Labor Relations Board concerning the recent certification elections.

There were no OSHA-recordable injuries during the month.

B. Major Problem Areas and Solutions

1. Leaky High-Pressure Valves in Hydrogenation, Section 600

   Valve leaks have delayed start-up activities in the Hydrotreating Section by 30 days. Of the 103 Conval process block valves in Section 600, 32 required repairs to correct packing, bonnet, and/or seat leaks. Of these, 24 have been repaired; of the remaining valves, it now appears possible that two will have to be completely replaced. Problems with the reused Edwards block valves in similar service have been relatively insignificant to date.

I. OPERATIONS

B. Major Problem Areas and Solutions (Continued)

2. Slow Heat-Up Rate and Low Temperature in Solids Separation, Section 300

The addition of insulation to the exposed manways, vessel heads, and flanges increased the overall temperature profile in the Solids Separation Section by only 10-15°F. The primary settler, F-B331, reached only 515°F, more than 100°F below design.

Solids separation was apparently satisfactory during the brief period in which extract was being processed. Continental Coal Development Corporation's recent bench-scale experience strongly suggests that settling temperatures must be increased toward 600°F as coal-derived liquids replace the start-up solvent. Further system modifications to improve Section 300 temperatures must be developed.

3. Recurring Extractor Seal Failures, Section 200

Both the top and bottom extractor agitator shaft seals failed during October. In each case the roll pin which positions the stationary seal member sheared. The subsequent rotational movement damaged the bellows. Hardened pins were installed with the replacement seals. Also, the differential thermal expansion of the shaft and extractor shell was less than expected. The seal components were repositioned closer together to increase the loading across the seal faces at operating temperatures. This change should result in the required force being applied to the sealing faces.

4. Failure of the PTI-54 Pump Coating

The PTI-54 ceramic lining in J-A313A, the primary hydroclone feed pump, was found to be severely damaged. An investigation into the causes of the failure has just started. There are sixteen Pacific pumps in the pilot plant which are lined with PTI-54, an alumina/titania material.

5. Extract Handling Difficulties in Solvent Recovery, Section 500

The extract granulator proved inoperable during the first attempt to slurry extract to the storage pond. All other means for removing the bottoms product from Section 500 plugged with extract in less than a day. Part of the problem resulted from the extract-to-solvent ratio rising above 8:1. The extract-rich streams solidified at points where metal temperatures were not maintained due to deficiencies in insulation and/or tracing.

I. OPERATIONS

B. Major Problem Areas and Solutions (Continued)

5. (Continued)

Prior to the next run, the granulation system will be modified to approximately the same configuration which was used successfully during Cresap I. Insulation and tracing will be supplemented where problems occurred. If practical, some points of hot-flush solvent addition will be modified to permit more efficient preuse heat-up and post-use purging of lines and equipment which process extract-rich streams. Operating conditions will be modified, reflecting the limited information collected to date, to maintain the initial extract-to-solvent ratio at 8:1, or less, in the extract-rich streams.

C. Progress

During a 20 hour operating period 8,000 pounds of coal were fed and 2,500 pounds to 3,000 pounds of extract were produced. This indicates a 35 percent to 40 percent depth of extraction compared to the 70 percent sought. The low conversion resulted from low extractor temperatures: 725°F inlet, 640°F outlet. Previous Consol experience with Sunoco FS start-up solvent at 640°F resulted in 35 percent conversion. The spray solvent heater, C-A204, was not working; the addition of cold spray solvent lowered the temperature at the top of the extractor to 605°F.

Based on very limited data, the primary settler rejected 95 percent of the feed solids to the underflow. The overflow solids content was generally less than 1 percent.

Solvent recovery was as expected; however, the processing conditions selected for the flash still system resulted in a very low solvent concentration in the bottoms product. Deficiencies in tracing and insulation contributed to the pump and line pluggages which terminated the run. Apparently no material was produced which boils between the Sure-Sol end point (560°F) and the initial boiling point of the extract (825°F).

Section 200 - Coal Extraction

Coal slurry was processed for the first time on October 11. After one hour of operation at a slurry feed rate of 4,000 lb/hr, a gasket failed on the pressure transmitter, PT-208-1, requiring an immediate shutdown. Upon attempting to restart, the slurry preheater, B-A201, was found to be plugged. Attempts to dislodge the blockage failed;

I. OPERATIONS

C. Progress (Continued)

Section 200 - Coal Extraction (Continued)

the middle coil in B-A201 was replaced. During the shutdown, inspection of the J-A202A and B slurry feed pumps revealed that the disc valves were pitted and did not seat properly. Ball checks with tungsten carbide seats have, therefore, been installed in place of the discs in both pumps.

A second slurry run was begun on October 18 at the same conditions. Twenty hours later coal feed was discontinued because of plugged lines in Section 500.

Repairs to the extractor agitator, L-B205, shaft seals were made twice this month. The details are discussed under Test 1800, below.

The on-off controller for the solvent heater, C-A204, failed after 18 hours of operation and was replaced with a silicon controlled rectifier proportional controller. Thermocouple leaks on C-A204 prevented the heater's use during the 20 hour run.

The tip of the plug in LCV-201-1, slurry let-down valve, broke off during operation, apparently because of a misalignment. The trim in the valve has been replaced.

Section 300 - Solids Separation

Both integrated and closed loop circulating modes, at normal operating conditions, were conducted routinely this month. Operation on extract did not present any abnormal problems. During the period when extract slurry was being processed, solids concentrations in the settler overflow varied between 2.62 percent and 0.26 percent with a median value of 0.31 percent. Bottoms from the underflow receiver (F-B330) were disposed of routinely in the carbonizer.

High liquid levels are present in all vessels as a result of the shutdown of Section 500. Solvent accumulates in the system from equipment seal flushes, solvent purges, and sampling. Excess inventory will be temporarily stored in the F-A1107 tanks, until Section 500 is back on stream. Hot, closed loop circulation will be maintained until the restart of Section 500.

Insulation of numerous vessel manways and flanges was accomplished to reduce process heat losses. Settler temperature was increased to 515°F from a previously attained maximum of 470°F.

I. OPERATIONS

C. Progress (Continued)

Section 300 - Solids Separation (Continued)

The flow control valves, FCV-322A and B, on the line feeding underflow receiver (F-B330) bottoms to the carbonizer (D-A801) were found to be undersized. They have been temporarily modified for split range operation. Replacement valves are scheduled for delivery in November.

Section 500 - Solvent Recovery

The section was switched from cold solvent circulation and placed in a solvent boilup, hot stand-by mode several days prior to coal addition in Section 200. The accumulated solids from Section 300 feed were drummed out, with the heavy solvent ends.

The first coal addition, which lasted one hour, resulted in the concentration of a heavy material in the E-A501 flash still loop. This material was drummed out when laboratory analysis indicated it was not a true extract, and the section was returned to the hot standby mode.

The second coal addition allowed unsteady state operation on extract for approximately 30 hours before plugging of lines and equipment forced a shutdown. The first pluggage occurred about ten hours into the run when the J-A502 flash still bottoms pump was lost due to less than 15 percent solvent in the extract material being processed. The conditions of 4 psia and 475°F at the E-A501 overheads were too extreme for the boiling range material being produced in Section 200. Operations were continued at conditions of 4.7 psia and a maximum temperature of 443°F. The heavy ends were drummed out through the E-A501 bottoms sample station, due to plugging of the drum-out station, until granulation was attempted. Efforts to granulate the extract stream were unsuccessful, and were aborted after the granulator feed line and the granulator system plugged. A failure of the LT-501 ball float flash still level indicator occurred about the same time as the granulator pluggage, allowing overfilling of E-A501, forcing the pilot plant to be shut down for cleanup of Section 500.

It appears that the granulator tank, F-A501, was either overfed with the extract stream, or the syphon, G-A501, did not work as expected and failed to granulate the extract. A redesign of this system has begun.

I. OPERATIONS

C. Progress (Continued)

Section 500 - Solvent Recovery (Continued)

Thirty-one drums of flash still bottoms were generated during the brief coal operation, and a sufficient number of bottoms samples was taken to allow calibration of the laboratory liquid chromatograph.

Section 600 - Hydrogenation

Leak testing of the Conval valves continues at 600 psig as repairs are completed. The Conval valve leaks are caused by damaged seats, defective packing, and damaged machined parts. Initial repair work was defined as:

1. Seat refinishing: 18 valves
2. Repacking: 28 valves and
3. Replacement of parts: 7 valves

General repair procedures were reviewed with the Conval representative. Current work consists of repacking the stems with Garfite 100 (top and bottom rings) plus valve seat lapping. Repair and retesting of the valves has been hampered by the continued presence of blasting grit and other foreign matter. A temporary pump and filtering loop will be set up to flush the valves and process lines of the debris.

Operation of the JC-A601A hydrogen recycle compressor, using nitrogen, has been carried out by all shifts at 3000 psig. The JC-A601B hydrogen recycle compressor experienced an internal knock during a test run. It remains in the shop pending replacement of the rod needle bearings and relocation of the lube oil reservoir heater.

The new weir plate for F-A602 high pressure separator was temporarily installed and leak tested. A permanent Viton gasket for the weir is on order.

The section nuclear level devices were chedked out by the service representative and made operational. Shop testing of forty-five section relief valves is continuing. Of several valves tested to date, only two relieved at the specified set pressure. Remaining control valve modifications deferred by construction were completed.

I. OPERATIONS

C. Progress (Continued)

Section 700 - Fractionation

Solvent has been charged to the system and is circulating cold, in a closed loop. The purpose of the circulation is to prevent possible freeze-up and check equipment operability. Operation has been routine.

Section 800 - Low Temperature Carbonization

Several days of sustained operation without the start-up heater were attained. Both the upper and lower carbonizer feed nozzles were utilized. The upper nozzle was used more frequently as it can be cleaned without removing the char bed from the vessel. Nozzle pluggage occurred three times, each time as a result of gasket material blocking flow to the nozzle.

The char quench system was used to dispose of excess char inventory to the char pond; its operation was routine. The flow of char through the char cooler, C-A803, could not be controlled.

A low flow problem in the slurry line to Section 300 was cured by increasing the control valve trim size. Erosion resulted in the replacement of spray nozzles and hydroclone separator supplying flush liquid to the seal of the tar quench pump, J-A806A. Additional spare separators were purchased and have been sent out for internal chrome plating.

Section 900C - Hydrogen Compression

The JC-A900A and B hydrogen makeup compressors were operated on nitrogen at pressures exceeding 3300 psig. Major leak repairs around the compressors are complete. All 600 Section operating technicians have some operating experience on the two compressors. The compressors are on standby with nitrogen valves installed in preparation for 600 Section leak checking at 2000-3000 psig.

Except for PV-951, control valve modifications deferred by construction have been completed.

Section 1000 - Utilities

Section 1010 - The boilers experienced flame-outs this month when condensate from the flare header backed through a vent line into the fuel gas line at the boiler. The vent line was disconnected from the flare header and now discharges directly to atmosphere, as per the MFD.

I. OPERATIONS

C. Progress (Continued)

Section 1020 - The cooling tower water distributors were cleaned. They will be recaulked to provide better spray patterns. This problem is not affecting plant cooling because of cool weather.

Section 1030 - Instrument air fluctuations in the main control panel were caused by undersized regulators; larger capacity regulators were installed.

Section 1040 - Fuel oil use in the boilers was replaced with less expensive natural gas, after the Hope Gas Co. advised that any curtailment this winter would not be affected by gas usage at this time.

Section 1050 - The electrical and instrument checkout of the emergency fuel gas system was completed.

Section 1100 - Offsites

Section 1110 - The process tanks were set up to handle virgin Sure-Sol 180 as if it were a donor solvent in Tanks F-A1106A, B, and C and to handle used Sure-Sol 180 as if it were recycle solvent in Tanks F-A1101A and B.

Section 1120 - The liner in the extract pond, F-A1122, is more likely to withstand solvent attack than the liner in the char pond, F-A1121; therefore, the initial char product, which could have contained traces of solvent, was sent to the former.

Section 1200 - Environmentals

Attached are the results of wastewater analyses for the past month. All parameters are within proper limits with the exception of one hexavalent chromium analysis. No reason for this excursion could be found.

D. Test Programs

General

Computer programs to store and report test data from log sheets are being developed. To date the filing program has been completed. Actual data are being filed to build a data base for program trouble-shooting. The display program is in the checkout stage and the report generator program is pending.

I. OPERATIONS

D. Test Programs (Continued)

General (Continued)

A laboratory study was initiated to determine if the pluggage problems in the seal oil system experienced last month were related to moisture in the Sure-Sol 180. Thus far all samples, including a fuel oil control sample, have shown approximately 3 mpy corrosion. The Sure-Sol spiked with water has consistently shown pitting, whereas the fuel oil and nonspiked samples have demonstrated only general weight loss.

Test 1100 - Pumps

Prerun inspection reports were completed for all but one centrifugal pump. The prerun inspection is pending for underflow pump, J-C303, which was recently returned from the Vendor.

All processed film from prerun inspections has been cataloged and labeled. Duplicate prints were forwarded to SCD.

The extractor charge pump, J-A202B, experienced a lubrication failure for the C cylinder packing. This failure caused extreme wear and scoring of the PTI-54 ceramic coating on the plunger. Inspection of the disc valves in both pumps, J-A202A and B, indicated roughness and pitting of the flat seating surfaces; their exposure to coal slurry was approximately one hour. Leakage past the valves is suspected. A post-run inspection of the disc valves and ports is in progress. The disc valves were replaced with ball check assemblies using high chrome balls and champhered tungsten carbide seating inserts. No pumping problems were experienced with the 10 percent coal slurry during the subsequent 20 hours of operation.

Test 1300 - Fired Heaters

Plugging of the middle pass coil occurred during the emergency dump of the slurry preheater, B-A201. Further information is presented in Section C, above.

Test 1500 - Filtration

Krauss-Maffei approved the use of a Mobil-therm product for the seal lubricant/cooling system. Warehouse addition requests for all lubricants required in the mechanical checkout procedure were issued. The checkout will take a minimum of three weeks, and will probably require additional personnel if undertaken at this time.

I. OPERATIONS

D. Test Programs (Continued)

Test 1700 - Instrumentation

The SCD test coordinating engineer visited the field to discuss the test program with the Liquefied Coal Test Engineer and perform some prerun inspection work.

Test 1800 - Extractor

The top seal of the extractor agitator, L-B205, failed on September 27. Inspection revealed that the 3/32-inch roll pin holding the stationary member sheared and lifted the stationary seat allowing leakage past the secondary packing. The bellows, seal faces, and packing (except the secondary) were in satisfactory condition. The flushing cup was slightly scored due to misalignment between the shaft and the seal gland. This resulted from a lack of perpendicularity betwen the seal gland and the shaft. The seal gland was machined to compensate for the misalignment and a hardened tool steel pin was installed to retain the stationary seat. These repairs did not stop the leakage. During the second disassembly the carbon seal face was lapped to correct out-of-flatness, but this repair was also unsuccessful. Effective results were achieved by raising the rotary seal member 1/2 inch to increase the pressure on the seal faces. This corrected a design error by Sealol, who had assumed that thermal expansion would be sufficient to compress the seat against the stationary face. The top shaft seal on L-B205 performed satisfactorily during both runs with coal.

On October 21, while Section 200 was on standby circulating hot solvent, the bottom seal failed. Inspection revealed that the bellows had ruptured and that the 3/32-inch roll pin on the stationary face was sheared. In addition, the bottom bearing was damaged by solids contamination. Repairs are in progress to replace the bellows, seal faces, packing, and bearing. In addition, the rotary member will be lowered 1/4 inch to provide more pressure at the seal face, compensating for the fact that thermal growth is less than predicted.

Test 1900 - Corrosion/Erosion

The Vendor-recommended modifications to correct the leaks in the test element holders were received. It is estimated that $5,000 to $7,000 would be required for additional parts.

I. OPERATIONS

E. Laboratory

The analyses of samples from the plant start-up were completed and reported in a timely manner.

A survey of the Modified ASTM D-1160 Vacuum Distillation Method used for samples containing extract and solvent was conducted. Responses received from Wilsonville (Catalytic), Tacoma (Gulf), and Lummus concerning their modifications to this method are being compared to the CCDC (Conoco) method to determine which should be used at this facility.

A safety film "Safety in the Chemical Laboratory," produced by Fisher Scientific Company, was shown to all shifts in the laboratory.

Samples of drinking water from the administration building are being forwarded to the West Virginia Department of Health every two weeks for free evaluation.

F. Safety and Health

There were no OSHA recordable injuries during the period.

The first joint meeting of the Central and Employee/Management Safety Committees was held during October.

The Central Safety Committee held their regular monthly meeting, also.

The final draft of the valve and line breaking procedure is being prepared.

Annual physicals and quarterly skin examinations continue.

The fire brigade members received training for the Emergency Procedure JSI.

G. Departments

Administrative

Staffing: Recruiting for Technical and Maintenance and Engineering Department vacancies has been quite unsuccessful the past several weeks. Intensified efforts continue to fill these openings. David Maxfield, a test engineer, has resigned effective October 28; Tom Chaney has joined the Mechanical and Engineering Department as a shutdown planner. Edward Smith also joined the Maintenance and Engineering Department as a warehouse clerk.

I. OPERATIONS

G. Departments (Continued)

Administrative (Continued)

Personnel: The International Chemical Workers Union has filed five objections to the result of the representation election held last month. To date, there has been no further information from the National Labor Relations Board.

Technical

The major Process and Shift Engineering efforts continue to be supportive of establishing integrated front end operations. Sampling requirements are being reviewed with an eye towards obtaining the most meaningful information from a minimal effort during the shake-down operations. Initial concerns will be to develop a nondetailed material balance using the major streams, and to develop indicators on the performance of each process section.

Hardware problems have developed in the computer which are preventing system preservation. The test data entry program is operational; test data entries are current from October 17. The work order system and the lubrication scheduler are operating normally. A contract programmer is correcting software errors in the warehouse system.

Maintenance and Engineering

Work is continuing on approximately ninety unpoured conduit seals identified during a plant-wide survey.

Steam tracing and insulation of equipment for proper winterization continues to receive major emphasis.

The two new water softeners for the hygienic program were installed and made operational.

A large number of routine instrument work requests are being handled satisfactorily by using the short work form.

Several plant interviews were held for the positions of designer/ draftsman. This position and that of mechanical engineer remain open.

Steve Schaefer arrived at Cresap from SCD for temporary assignment in plant engineering.

I. OPERATIONS

G. Departments (Continued)

Maintenance and Engineering (Continued)

Work order activity over the past month is as follows:

	Mech.	E&I	Total
W. O.'s Opened	103	43	146
W. O.'s Completed	126	82	208
W. O. Histories	14	29	43
W. O.'s Closed	---	--	95
B/M's Issued	24	--	24

II. TEST PROGRAM

A. General

1.0 See Part I, Section C for additional details.

B. Tasks

1.0 Solvent Deashing

1.1 Objective:

To determine the process design for solids-liquids separation using the solvent deashing concept developed by CCDC, with verification of the design on both the bench scale and pilot plant scale bases.

1.2 Work Accomplished for the Month:

The draft of the section on process engineering studies prepared by Conoco Coal Development Company for inclusion in the final report was received and discussed with Conoco. The draft of the final report is being reviewed. Preliminary comments were also discussed with Conoco.

1.3 Problems Encountered and Solutions:

A significant temperature drop across the solids separation section was apparent when the unit was on internal recycle. It is partly caused by mixing cold recycle with hot feed. Some insulation was added to reduce large heat losses. This is an abnormal operation which is anticipated to correct itself during integrated running.

1.4 Work Forecast for the Coming Month:

II. TEST PROGRAM

B. Tasks (Continued)

1.4.1 Return comments to Conoco on the final report draft.

1.4.2 Review Conoco's revised draft of the process engineering studies.

2.0 Liquid-Solids Mechanical Separation

2.1 Objectives - The overall objectives are:

2.1.1 To determine maximum filtration rates with various attractive candidate filtration equipment.

2.1.2 To evaluate mechanical design, configuration and reliability.

2.1.3 To analyze the operation economics of the various filtration schemes with various coal liquefaction process streams.

2.2 Work Accomplished for the Month:

2.2.1 Completed final test procedure for Mechanical Checkout of Horizontal Leaf Filter. Procedure distributed to field and ERDA. Completed second draft of Rotary Drum Test Procedures and sent to field for review.

2.3 Problems and Solutions:

2.3.1 The spare parts returned to Ametek for replacement have not been returned.

2.4 Work Forecast for Coming Month:

2.4.1 Complete review and approval of all test procedures.

3.0 CSF Process Extractor Improvement

3.1 Objectives:

3.1.1 Evaluate process variables inherent to the CSF Process Extractor mixer.

3.1.2 Determine the performance capabilities of mixer mechanical components.

3.1.3 Evaluate mechanical and nonmechanical Extractor design configurations with respect to mechanical reliability, process capability and scale-up potential.

II. TEST PROGRAM

B. Tasks (Continued)

3.2 Work Accomplished for the Month:

3.2.1 Coordinated arrival of single mechanical seal Vendor representative on site to evaluate mechanical seal problem. (See Problems and Solutions)

3.2.2 Requested written submission of quote for mechanical vibration probes.

3.2.3 Discussed bearing design changes with Vendor and implemented this information during shutdown period.

3.2.4 Evaluated preliminary operating data.

3.2.5 Recorded all events, observations and conclusions relating to Extractor operations during this time period.

3.2.6 Provided aid to LCDC and SCD field personnel during Extractor operations.

3.3 Problems and Solutions:

Operation of the Extractor continued uneventfully during this time period until sudden failure of the upper single mechanical seal occurred. Teardown of the seal revealed mild to severe galling in various parts related to the seal. The cause was suspected to be due to misalignement. Subsequent checks revealed higher than normal perpendicular and parallel misalignment. Corrections were made and a rebuilt seal was fitted. Upon pressurization the seal held until 350 psig at which time a steady leak occurred. Upon slow depressurization the leak stopped at 0 psig. This cycle was repeated several times. Since no reasonable explanation was available the single mechanical seal Vendor was called in. His investigation revealed that the seals needed to be reset to increase the seal face loading. This was accomplished and operation was then restarted.

3.4 Work Forecast for the Coming Month:

3.4.1 Continue evaluation of alternate material for mechanical vibration probe tip.

3.4.2 Coordinate data transmission from LCDC.

3.4.3 Prepare subcontract for single mechanical seal site visit.

3.4.4 Evaluate daily reports and data from field. Continue update of home office Test 1800 General Record.

II. TEST PROGRAM

B. Tasks (Continued)

3.4.5 Provide assistance to LCDC and SCD field engineers during Extractor operations.

4.0 Activity in all other test elements was of a minor nature.