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PETROLEUM PROCESSING RESEARCH AND DEVELOPMENT AT THE FEDERAL DEPARTMENT OF ENERGY, MINES AND RESOURCES

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by

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ABSTRACT

In Canada, the federal department of Energy, Mines and Resources through its Canada Centre for Mineral and Energy Technology (CANMET) is actively supporting petroleum processing research and development in a blend of in-house and contract-out programs. This presentation outlines the research projects, describes the equipment and facilities and identifies the manpower and budgets which are available to support the research activities.

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INTRODUCTION

The Department of Energy, Mines and Resources, through its Canada Centre for Mineral and Energy Technology (CANMET), formerly called the Mines Branch, has maintained an uninterrupted interest in low-grade fuel resources for more than 50 years. Table 1 gives a summary of activities carried out by the Energy Research Laboratories from 1930 to the present.

In 1930 batch autoclave experiments were carried out to show the amenability of the hydrogenation process to the refining of Alberta bitumen. In 1933 a continuous hydrogenation pilot plant with a ten-litre reaction vessel for operation at 3000 psi pressure was installed and experiments were done on the hydrogenation of coal, peat, char and bitumen. This work was put aside during the war years and revived again in 1945; when hydrogenation experiments were done using bitumen supplied from the small, experimental, hot-water separation plant situated on the Clearwater River near Waterways, Alberta, set up by Dr. K.A. Clark of the Research Council of Alberta. The hydrogenation experiments at that time led conceptually to a complicated process involving distillation, vapour-phase hydrogenation, centrifugation and coking. It was postulated that coking of the entire bitumen stream would eliminate several steps and reduce capital costs, and to this end a "flash" coker was built in 1949 which could produce coke and coker distillate in a batch mode, or pitch and somewhat less distillate in a continuous mode. In the late 1940's, vapour-phase hydrodesulphurization of distillate oils and coker distillate was undertaken to complete the package for the coking process for Athabasca bitumen and to offer the alternative of hard pitch plus distillate to heavy-oil producers.

Work on the cold water separation process was started in the Mines Branch after a fire caused the shutdown of Abasand Oils Limited in 1945. Bitumen produced in a cold-water separation pilot plant built in 1949 was used in subsequent coking and hydrocracking studies. There has been renewed interest in cold-water separation and work on this process is proceeding under contract.

Early work in liquid phase hydrogenation and hydrocracking was performed in the 1960's. This was followed by many experiments in catalytic and non-catalytic hydrocracking on bench-scale and pilot-scale apparatus, which demonstrated that over 10% more liquid distillate product (weight or volume) could be obtained by hydrocracking than by the coking process. These experiments were of short duration to determine the effects of temperature, hydrogen partial pressure, LHSV and gas recycle rate on the parameters shown in group 1, Table 2.

The next steps in the development of the hydrocracking process involved the assessment of operability and the solution of technical problems regarding product and by-product separation and treatment as shown in Table 2.

Figure 1 gives statistics regarding pilot plant activity in recent years, The large increases in pilot-plant "hours on stream" since 1972 were a direct result of increased interest in operability. The improvements in 1976 and 1977 were due to increased staff, and the output in 1978 and later is expected to increase slightly as the technical staff gains in experience and a new data acquisition system is installed. Since 1976 there has been a shift from mined bitumen to heavy oil experiments. In 1976, 96% of the processing hours were with mined bitumen feed. In 1977 this figure was 64%, with the balance devoted to heavy oils and in-situ feed. A second pilot plant came on-stream in 1979.

Research Planning

Federal energy research in Canada is not concentrated in the hands of a single department, but is planned and coordinated by an Interdepartmental panel with representation from the following federal departments and agencies:

Permanent members:

Energy, Mines and Resources Canada (EMR)
National Research Council of Canada
Ministry of State for Science and Technology
Transport Canada
Treasury Board
Fisheries and Environment Canada
Supply and Services Canada
Department of Industry, Trade and Commerce

Associate members:

Agriculture Canada
*Atomic Energy Control Board
*Atomic Energy of Canada Limited
Department of Consumer and Corporate Affairs
*Central Mortgage and Housing Corporation
Department of Indian and Northern Affairs
Department of Finance
*National Energy Board
*Petro-Canada

*(government-owned corporations)

A secretariat for the panel is provided by EMR, which is also the lead agency for conservation and fossil fuel research. The principal organization carrying out fossil fuel R & D studies is CANMET.

The federal government's activity in energy research and development is necessarily a compromise among the principles of allowing the market place to look after itself, of ensuring that Canadians have access to needed technology, of deciding when new technologies will become operative and of restraining the growth of federal government spending. The rationale for any particular research performed or funded by the federal government tends therefore to be based on one or more of the following:

- the technology is too expensive and the payoff too uncertain to attract the private sector (e.g., methane from biomass).
- long-established federal government capability exists and is not available elsewhere under practical circumstances (e.g., CANDU nuclear test reactors).
- the federal government must maintain a high level of expertise as backup to policy decisions (e.g., transportation of fuels).
- the technical problems are uniquely Canadian (e.g., the oil sands).
- a need exists to spread technology across a sector of the Canadian economy (e.g., support research of the Canadian Electrical Association).
- a need exists for at least a minimum commitment to maintain access to developments in other countries (e.g., nuclear fusion).

It is also necessary, of course, that all research meet certain basic requirements of federal science, such as good management, favourable conditions for technology transfer or implementation, and conformity to federal energy policy goals.

Although the federal government conducts much research on its own, current policy promotes the contracting-out of federal research to the private sector when feasible. In this way, Canadian industry can participate to the fullest extent possible in meeting national goals in science and technology. About 40% of the total federal expenditures on energy R & D are spent extramurally and continuing efforts are being made to increase this figure.

Universities also carry out federally-funded research. For mission-oriented research they are eligible for contracts issued by various departments and agencies. The new Natural Sciences and Engineering Research Council provides continuing support for fundamental studies in all scientific and technical fields. Joint programs involving the public, private and academic sectors are common, and Canada participates in international joint efforts such as those of the International Energy Agency.

CANMET Energy Research Program

The CANMET energy research program deals mainly with fossil fuel and conservation technology, with lesser emphasis on renewables, nuclear, electricity, energy storage, and energy transportation. In addition to the usual goals associated with R & D, CANMET helps provide the technical knowledge base required by policy-makers in EMR. The program is implemented by a matrix management system (Fig. 2), in which line management implements programs planned by program offices. In a sense the program office plays the role of the "client", with the laboratory divisions being the "contractors"; for contract programs, the external contractor is then essentially a sub-contractor.

In 1980-81, the CANMET energy research budget totals almost 16 million dollars, including salaries. About 25% of this total is spent on external contracts.

Petroleum Research

Most of the research on conventional petroleum and natural gas technology in Canada is carried out by industry, and EMR's role is to ensure that the gaps left by industrial interests are filled.

Most of CANMET's petroleum research is directed towards the oil sands and heavy crude oils of Western Canada, in which there is also heavy industrial involvement. These constitute one of the largest concentrations of fossil fuel resources in the world, and are now under development with two commercial plants operating and two more planned.

CANMET's effort has centred mainly around development of the CANMET Hydrocracking Process to upgrade bitumen and other heavy petroleum feedstocks. This patented technology has been turned over to Petro-Canada for further development and commercial application.

Equipment Facilities

The equipment facilities to carry out the research programs range from small scale analytical instruments to one barrel per day high pressure pilot plants. The details of the equipment facilities are listed in Table 3.

A. Pilot Plant

The first hydrocracking pilot plant was completed in 1965 and was improved over the years. This plant is still operational in the laboratories along with a new one barrel per day plant completed in August 1979. Both of these plants are designed to operate at high pressures and temperatures and to handle viscous feedstocks and slurries. In the design and construction of these plants, specific attention was given for safety to include explosion proof walls, gas alarms, exhaust fans and fresh air intakes, etc.

The pilot plants have been well designed to enable continuous operation over extended periods of time. The high pressure pilot plants have gone through several modifications and a broad base of expertise has been developed on high pressure equipment performance and selection. The recent successes in CANMET Hydrocracking Process development are partly because of reliable equipment development and in-house expertise on equipment design and modifications. The operators are trained in-house because of specialized nature of equipment and operations.

A coal liquefaction pilot plant is also currently in operation and proposals are being studied to convert one of the existing large one barrel per day pilot plants for co-processing of coal/heavy oil and for coal liquefaction.

A pilot scale six barrel per day vacuum distillation column is available for preparation of feedstocks and distillate fractions. Several distillations have been done in this distillation column under contract to support research activities in outside industry.

B. Bench Scale

The bench scale continuous flow and autoclave facilities are available for catalyst development and feedstock evaluation. Extensive facilities are also available for catalyst preparation and surface characterization. Hydrotreating units are available to evaluate secondary refining of distillate fractions to meet synthetic crude specifications.

Small scale distillation equipment such as equilibrium flash still, microbalance system to evaluate pitch gasification, plastic models for reactor residence time distribution, are also available to support process research and development activities.

C. Analytical

The analytical facilities include routine ASTM and non-ASTM analyses as well as special instrumentation for characterizing heavy molecular weight compounds. Special instruments are used for analyzing gases, liquids and solids. Other groups in different sections of CANMET help in the characterization of reactor liquid and solid samples using instruments such as electron microprobe and scanning electron microscopes.

In-House Projects

The in-house projects were selected in order to develop methods for maximizing liquid yield from Canadian fossil fuels. In recent years, CANMET has made rapid strides in the development of a hydrocracking process for upgrading heavy oils and bitumen. This process, called CANMET Hydrocracking Process, is being commercialized by Petro-Canada. Several of the projects listed in Table 4 are in support of this commercialization program. The extensive research and development work for the past 15 years in the hydrocracking processes has resulted in several patents, publications and confidential reports. A comprehensive technology package has been developed for different feedstocks, operating conditions, catalysts and product qualities. Fig. 1 clearly shows the significant increase in activity for the last few years in CANMET pilot plant operations. Currently, pilot plant work is continuing at a high level of activity to obtain more information for scale-up and design and to support commercialization activities.

The bench scale work is on-going to evaluate new feedstocks from potential customers for the technology. Support studies are underway on pitch utilization and characterization of feedstocks, hydrocracked products, catalysts and reactor samples.

Research and development in catalysts have resulted in low cost catalysts and fouling resistant catalysts. These catalysts will also be useful for co-processing coal and bitumen and for liquefaction of coals. Some experiments have already been completed in the co-processing of coal and bitumen. Methods have been developed to test catalysts using rapid catalyst deactivation techniques. Catalysts with different pore sizes have been tested to develop optimum pore catalysts. In-house work in the past few years on catalyst surface analysis has been started using ESCA.

Rapid hydrocarbon characterization methods have been developed for polynuclear aromatics and special adsorbents have been developed for removal of nitrogen compounds. Work is continuing for improved analytical methods for characterizing bitumen and synthetic fuels by liquid chromatography.

Contract Program

A separate contract program is in effect for coal in addition to the one for oil and gas. The contracts in oil and gas were selected to improve data base for the CANMET Hydrocracking Process along with economic analysis of competing upgrading processes (Table 5). Contracts were used to get the best expertise from industry and universities and data were gathered on solubilities, pitch gasification, BTX potential of products, properties of products, secondary refining conditions, co-processing of coal/bitumen, compatability of products and pitch utilization. Contracts are also given to obtain support data for scale-up and design of the CANMET Hydrocracking Process.

Industry Interaction

The research and development programs in the Synthetic Fuels Research Laboratory have always been planned to encourage the participation of industry. Recently, since the initiation of the commercialization of the CANMET Hydrocracking Process, the industry interaction has increased significantly. In the past few years, the research laboratory has done contract work for companies like Imperial Oil, Petro-Canada, GCOS (Suncor), Gulf and Amoco.

The contract program in SFRL also enables a close contact with industry. The interaction with industry helps to maintain an up-to-date awareness of problems in petroleum processing and determines the direction of energy research in the future. Contacts have also been maintained on a regular basis with government organizations such as AOSTRA, ARC and Petro-Canada. CANMET staff regularly visited several research establishments and oil companies to keep a close contact with their developments.

Future Synthetic Fuels R&D at CANMET

The major objectives of our program will continue to be the development and/or improvement of technologies for increasing the quantities of liquid fuels available to the economy as well as to ensure adequate qualities of the synthetic liquids for use in transportation. In the case of the latter, particular emphasis will be given to diesel fuel.

It is difficult to attempt to predict the amounts of additional financial and human resources that will be allocated to our Synthetic Fuels program. The human resource allocations are of special concern to us. There are other complicating factors such as the government's policy to have as much work done in the private sector as possible and also regional impacts which are of great importance to our political masters.

In our planning the major thrusts are shown in Table 6.

Continued improvements will be made in the hydrocracking technologies. These improvements will involve reduction in operating costs and adaptations to deal with various feedstocks as required. Also, attempts will be made to increase the middle distillate fractions for diesel fuel production through research in engineering and on additives. It is envisaged that the future activity in hydrocracking will be at a lower level than at present.

Large efforts are being envisaged for combining hydrocracking with coal liquefaction - co-processing. The feasibilities for obtaining additional amounts of liquids during hydrocracking through contributions from added coals will be studied in great detail. This is a logical extension of the hydrocracking research in particular as coal is used for additives during hydrocracking. This technology, if feasible,

is well suited for application in the provinces of Alberta and Saskatchewan where bitumens and heavy oils are in close proximity to coals. In this report it might also have relevance to Venezuela. In eastern Canada we have considerable residual materials from imported oils and the possibility of upgrading these residuals during co-processing with coals will also be investigated.

It should be mentioned that the investigations of co-processing would be expected to yield considerable information for improving the CANMET Hydrocracking technology.

The other major thrust is the secondary upgrading of syncrudes for motor uses with emphasis on diesel fuel. These syncrudes from bitumens/heavy oils are considerably more difficult to upgrade than the conventional oils. For coal-derived liquids the situation is much worse. Therefore co-processed products will be difficult to upgrade.

The upgrading of these synthetic crudes to diesel fuels of satisfactory quality at acceptable costs is a major objective in our future planning. The challenge is great and requires much basic research in catalysis, characterization and engineering. It is our intention to involve participants from the private sector, provincial government agencies and universities as shown on the slides. It is believed that the participating industries would be those interested in secondary upgrading and those interested in catalyst manufacturing.

It should be mentioned that there is relatively little catalyst manufacturing in Canada and that this program could boost activity in this area. In addition some companies would be involved with no special interest except to perform R&D under contract.

Considerable interest is expected from provincial government agencies in particular AOSTRA. Obviously the basic R&D should involve universities and possibly the establishment of groups dedicated to studying the upgrading problems might be considered.

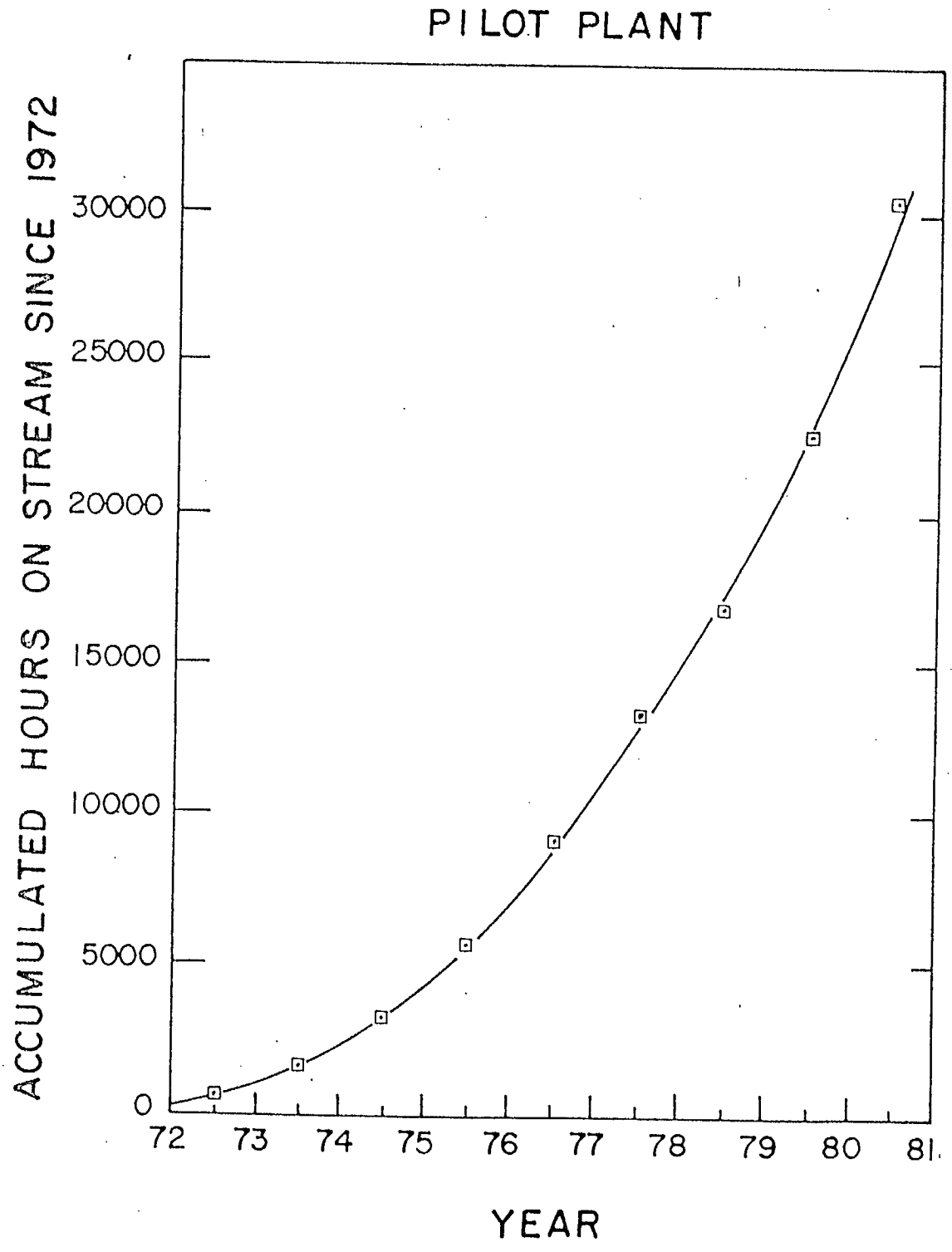
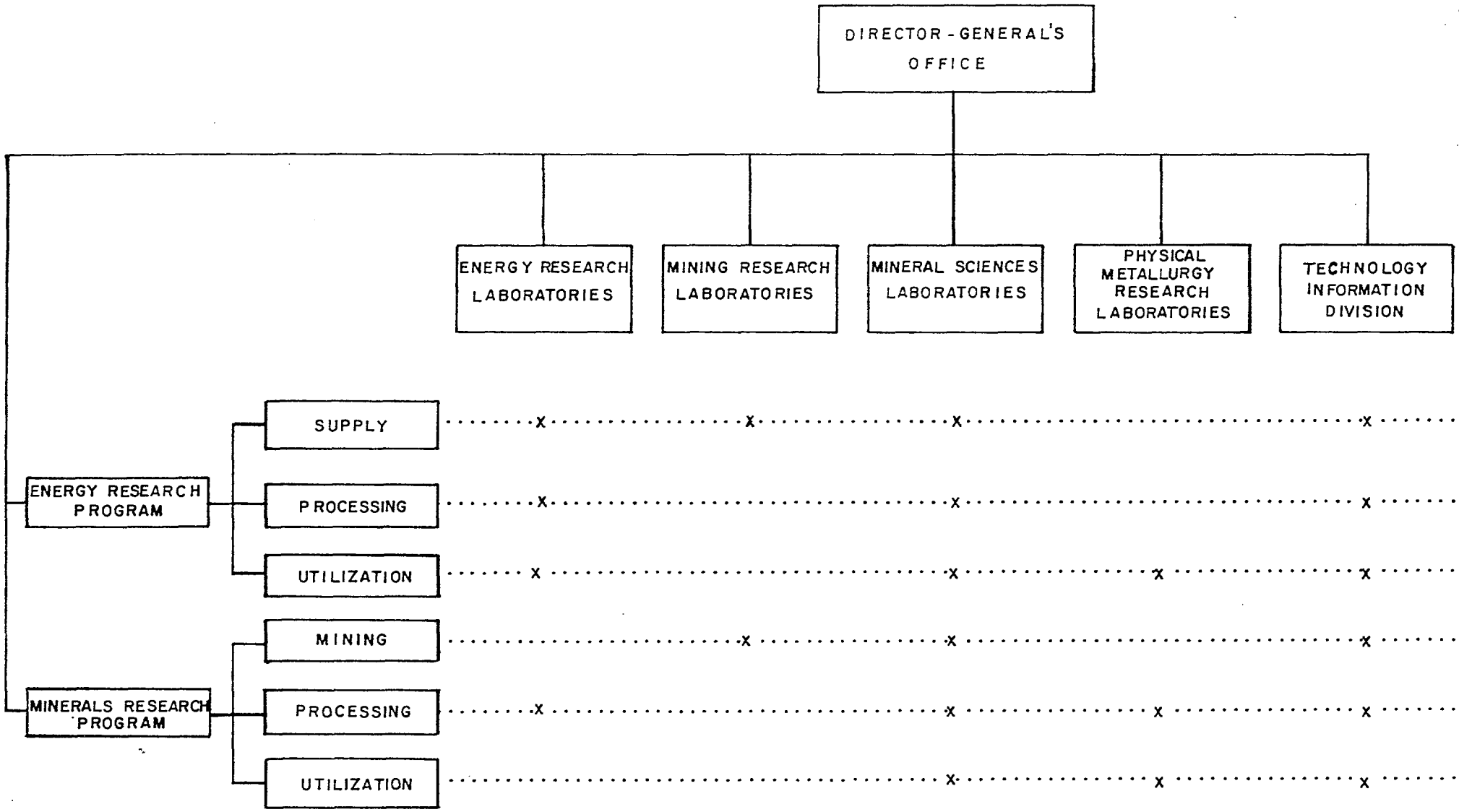


Fig. 1 - Hydrocracking Pilot Plant Operations since 1972



Matrix management system used at CANMET,

The laboratory division implement programs planned by the Program Offices

Table 1

Divisional Activities in Petroleum Processing

Division Name	Activity	Year
Fuels (Mines Branch)	Hydrogenation of Athabasca bitumen;	1930
	Liquefaction of coal, peat, char and bitumen;	1933
	Coal liquefaction;	Late 1930's
Fuels and Mining Practice	Vapour phase hydrodesulphurization of distillate oils and coker distillate; Cold water separation process;	Late 1940's and 1950's
	Hydrocracking of residuum;	Early 1960's
Fuels Research Centre	Hydrocracking of Alberta bitumen; Catalytic hydrocracking;	Late 1950's
Energy Research Laboratories (CANMET)	Hydrocracking of residuum, bitumen and heavy oils; Hydrogenation; Hydrodesulphurization; Cold water separation process;	Late 1960's
	Coal liquefaction and gasification	1977

Table 2

Significant Parameters in Hydrocracking Development

Distillate yield	
Product distribution	
Pitch conversion	
Hydrogen consumption	Group 1
Hydrocarbon gas make	
Sulphur removal	
Nitrogen removal	
Operation	Group 2
Product separation	
Product treatment	Group 3

Table 3 - Equipment Facilities

A. Bitumen Processing Section

1. Hydrocracking Pilot Plants:

- Two one-barrel per day pilot plants
  Reactor sizes: 1 1/2" x 13' , 2" x 8' and 5" x 14" CSTR, 3" x 36"

2. Hydrotreater:

- One litre per hour unit
  Reactor sizes: 2" x 2' and 2" x 4'

3. Distillation column:

- 6 bbl per day continuous distillation
  Column 10" x 22'
- 75 liter batch distillation column 4" x 48"
- Equilibrium flash still

4. Other units:

- 2 litre capacity autoclaves, plastic models, datalogger

B. Catalysis Research Section

1. Catalyst Preparation and Evaluation

- Continuous mix muller, grinder, batch tray drier, continuous rotary drier, batch tray calcining furnace, batch muffle furnace, BET surface area apparatus, mercury porosimeter, ESCA, surface acidity apparatus

2. Hydrocracking bench scale units

- Four continuous flow reaction systems (1" x 12" reactor), hydrogen storage system and catalyst life testing units.

3. Gasification:

- One batch microbalance system
  One continuous flow reaction system

C. Coal Liquefaction Section

1. Coal liquefaction pilot plant

- 500 g/hr slurry 3" x 10" CSTR

2. Hydrogenation unit - solvent preparation

Table 3 (Cont'd)

D. Analytical Section

1 Routine Analysis:

- Equipment for routine ASTM and non ASTM analyses such as viscosity, nitrogen, sulphur, pourpoint, CCR

2. Special Analyses:

- Mass spectrometer, GC/mass spectrometer
  C^{13} NMR and H^{1} spectrometer

E. Research on Bituminous Substances Section

- IR spectrophotometer, UV spectrophotometer
  high pressure liquid chromatography
  gas chromatography

F. Solids Evaluation Section

- LECO SC-32, atomic absorption and X-ray fluorescence

G. Other groups

- Petrographic reflective and fluorescence microscopes, microhardness testing, electron microscope, SEM, electron microprobe, corrosion evaluation, combustion equipment, coal and coke testing facilities.

Table 4 - In-house Projects

No.	Project Title
1.	Pilot Plant Operations - Thermal hydrocracking of bitumen and heavy oils
2.	Pilot Plant Operations - catalytic hydrocracking of bitumen and heavy oils
3.	Reactor modelling correlations
4.	Solid characterization
5.	Technology Transfer/Process Development commercialization
6.	Hydrotreating of distillate products
7.	Development of low cost catalysts
8	Development of fouling resistant and large pore catalysts
9.	Catalyst surface studies
10.	Pitch gasification studies
11.	Coprocessing coal/heavy oil
12.	Coal Liquefaction studies
13.	Coal Gasification studies
14.	Characterization of Bituminous substances
15.	Analyses of heavy oil and hydrocracked products.

Table 5 - CANMET Contracting-out Program

No.	Contract Title
1.	An Economic Study of Coal as a Make-up Energy Source in Oil Sands Processing
2.	Software Development for Pilot Plant Data-Logger
3.	Optimum Economic Generation of Hydrogen
4.	Electron Spin Resonance of Bitumen and Hydrocracked Products
5.	Measurement of Thermal and Transport Properties of Bitumen, Heavy Oils and Hydrocracked Products
6.	Coal as a Make-up Fuel in Tar Sands Upgrading
7.	PDU Evaluation of Commercial Scale Bitumen Hydrocracking
8.	BTX Potential of Naphtha Fraction Obtained by Thermal Hydrocracking of Bitumen
9.	The Assessment of the Potential of Synthetic Crude from Thermal Hydrocracking of Bitumen
10.	Pitch Gasification - Hydrocracking
11.	Hydrotreating of Distillates from Fluid Coking and CANMET Hydrocracking
12.	Economics of Hydrogen Production
13.	Feasibility of Using Bitumen as a Solvent for Coal Liquefaction
14.	Thermal Hydrogenation of Bitumen/Coal Slurries using Syngas
15.	Production of Standard Refinery Products from Synthetic Crude
16.	Gamma Interrogation Measurements
17.	Compatibility studies
18.	Chemical characterization of pitch
19.	Asphalt Production - Hydrocracked pitch
20.	Residence time distribution
21.	Coking characterization of pitch
22.	Hydrogenation catalysts
23.	Hydrodesulphurization of pitch.

Table 6

Future Synthetic Fuels R & D at CANMET

Major Thrusts

Continued Improvements in Hydrocracking Technologies

Coprocessing of Coals with Bitumens/Heavy Oils/Residuals

Secondary Upgrading of Syncrudes with Emphasis on Diesel Fuel