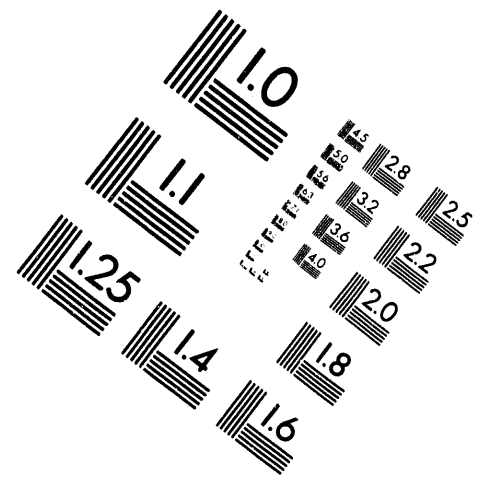
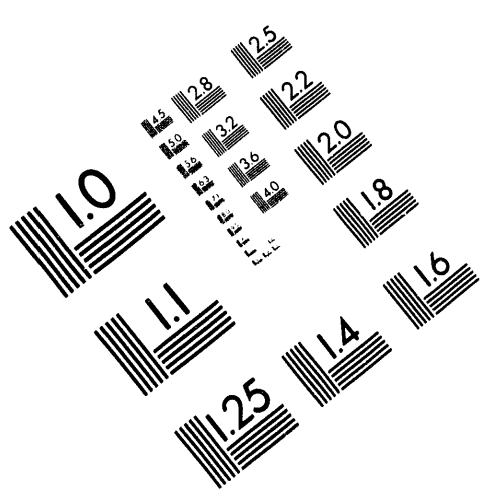




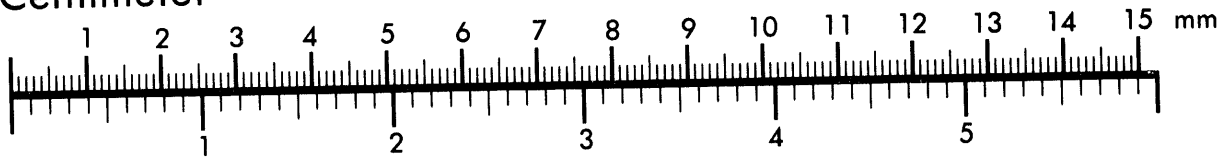
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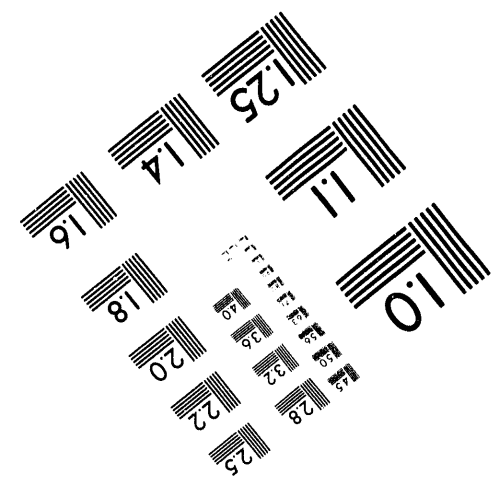
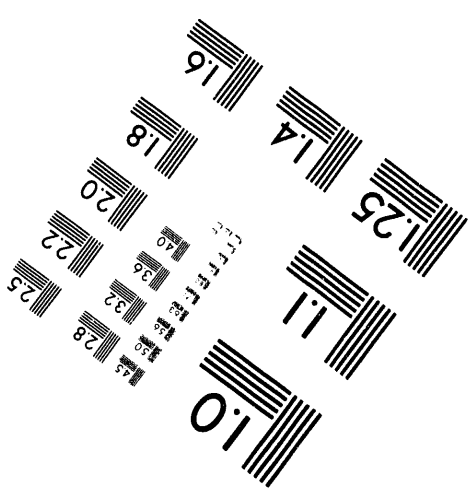
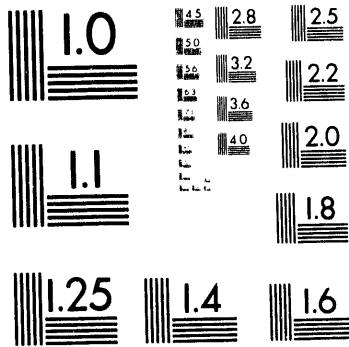
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1 of 1

CHEMICALLY ASSISTED *IN SITU* RECOVERY OF OIL SHALE

DE-AC22-89BC14479

The Department of Chemical Engineering
The University of Colorado at Boulder
Boulder, Colorado.

Contract Date: September 5, 1989.

Anticipated Completion Date: September 4, 1991.

Government Award: \$91,967.

Principle Investigator: W. Fred Ramirez.

Project Manager: W.D. Peters.

Reporting Period: April 1, 1990-June 30, 1990.

Objectives:

The objective of this work is to investigate, in the laboratory, the parameters associated with a chemically assisted *in situ* recovery procedure, using hydrogen chloride (HCl), carbon dioxide (CO₂), and steam (H₂O), to obtain data useful to develop a process more economic than existing processes and to report all findings.

Summary of Technical Progress:

This report covers the status of DOE project (Award Number DE-AC22-89BC14479; UCB Account Number 153-6529), specifically the milestone schedule status is reported. The research project supported is the investigation of a chemically assisted *in situ* oil shale extraction method, involving lower temperature and pressure than existing procedures, with the goal of developing an economically viable recovery method. The technical progress of the

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project is reported. The project status is that experiment preparations are underway, with most design work completed.

The progress of the project is that experiment preparations are underway. Reactor design, process design, and experiment design have been completed. The laboratory to be used has required extensive clean-up, and is nearly ready. Safety considerations are underway. Finally, an initial literature search has revealed some important aspects that need to be considered.

The reactor design is complete. The reactor will provide a constant temperature environment with a controlled gas pressure for a linear progression of the reaction interface through the shale core. The reactor will be constructed of stock 304 stainless steel. The reactor design consists of three flange plates (SS304 plate) sealing two cylindrical chambers (SS304 tubing) with silicon rubber gaskets. Threaded stock will provide the necessary compression on the gaskets. The first chamber will contain the oil shale core. The cylindrical shale core will be sealed to the chamber tubing with either a high-temperature-low-viscosity epoxy or with molten lead. Lead will be used only if the epoxies are found to be unsuitable. Because both sealing methods will cover both core faces, the ends will be sawed off to expose the oil shale core. The second chamber will serve as a gas mixing chamber where thermal convection will prevent the formation of gas phase concentration gradients and reactant depletion near the core face. This will maintain a high concentration gradient just within the oil shale, giving a high reactant flux into the core. The reactor temperature will be maintained by immersing the reactor assembly into a hot fluidized sand bath. The

"sand" is heated by electric heating elements, and the well-mixed property of the fluidized bed will ensure that the temperature within the bath is very uniform. The top flange, adjacent to the gas chamber, will be exposed to the atmosphere, providing a thermal gradient, whence thermal convection will provide mixing. A perspective drawing (Figure 1) and a cross-section drawing (Figure 2) of the reactor are included.

The design of the process is completed. Further information may necessitate modifications when more is known of the process itself. The reactant gases, HCl and CO₂, will be mixed in a pressure vessel (a Hastelloy C bomb) at ambient temperature and the appropriate pressure to achieve the desired molar ratios. Water will then be pump into the bomb, via an oil-water reservoir served by a Ruska volumetric pump. The bomb, residing in a second fluidized sand bath, will be heated to process temperature. The reactor gas chamber, after evacuation, will be heated also to temperature. The reactor gases will be introduced and the reaction(s) begun. Because the expected reaction stoichiometry is one mole gas evolved per one mole gas consumed, the reactant gas chamber pressure is expected to remain constant. Upon break-through of the reactant gases, the down-side of the core will be sealed-off. The remaining reactant gases (and product gases that have back-diffused to the chamber) will then be drawn off and sampled for quantitative analysis by gas chromatography for HCl, CO₂, H₂O, sulfur compounds, and light hydrocarbons. The gases will be cooled, condensed, and collected. To recover the released hydrocarbons, the core will then be flooded with hot water or steam. The secondary-type recover will be done at

a lower temperature than the reaction, with the temperature of the H₂O and of the core maintained by the fluidized sand baths.

Since very little is known about the actual reactions involved, the experiment design is in flux. Currently, we plan to start with core samples of approximately 2 cm in length, progressing to longer cores. It is expected that break-through time will be linear with respect to core length, because the reaction front progression is most likely diffusion limited.

An initial literature search has been informative. Recent work with kerogen has revealed by the use of controlled pyrolysis that the structure of kerogen is a highly three-dimensionally cross-linked carbon polymer (Schmidt-Collérus and Prien, Yen). It has been suggested that ozone, O₃, may be useful for freeing the kerogen by controlled oxidation of the cross-linkages (Young). The reactor design is ideal for such experiments, which may be useful to differentiate between various mechanisms.

The actual arrangement of the equipment, gas cylinders, sand baths, *et al* is close to completion. The University's Environmental Health and Safety department is being consulted, and any necessary changes must be accommodated. Heat shielding for the gas cylinders is one example of a possible requirement.

Currently, necessary equipment is being ordered. Reactor construction will begin soon. A source for oil shale has been found and we are awaiting shipment.

The project status is progressing smoothly. It is expected that trial reactions may be begun in September, 1990.

References:

- J.J. Schmidt-Collérus and C.H. Prien. "Investigations of the Hydrocarbon Structure of Kerogen from Oil Shale of the Green River Formation." Science and Technology of Oil Shale. T.F. Yen, editor. ©1976.
- T.F. Yen. "Structure Investigations on Green River Oil Shale Kerogen." Science and Technology of Oil Shale.
- D.K. Young, S. Shih, and T.F. Yen. "Mild Oxidation of Bioleached Oil Shale." Science and Technology of Oil Shale.

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Reactor Assembly Perspective View

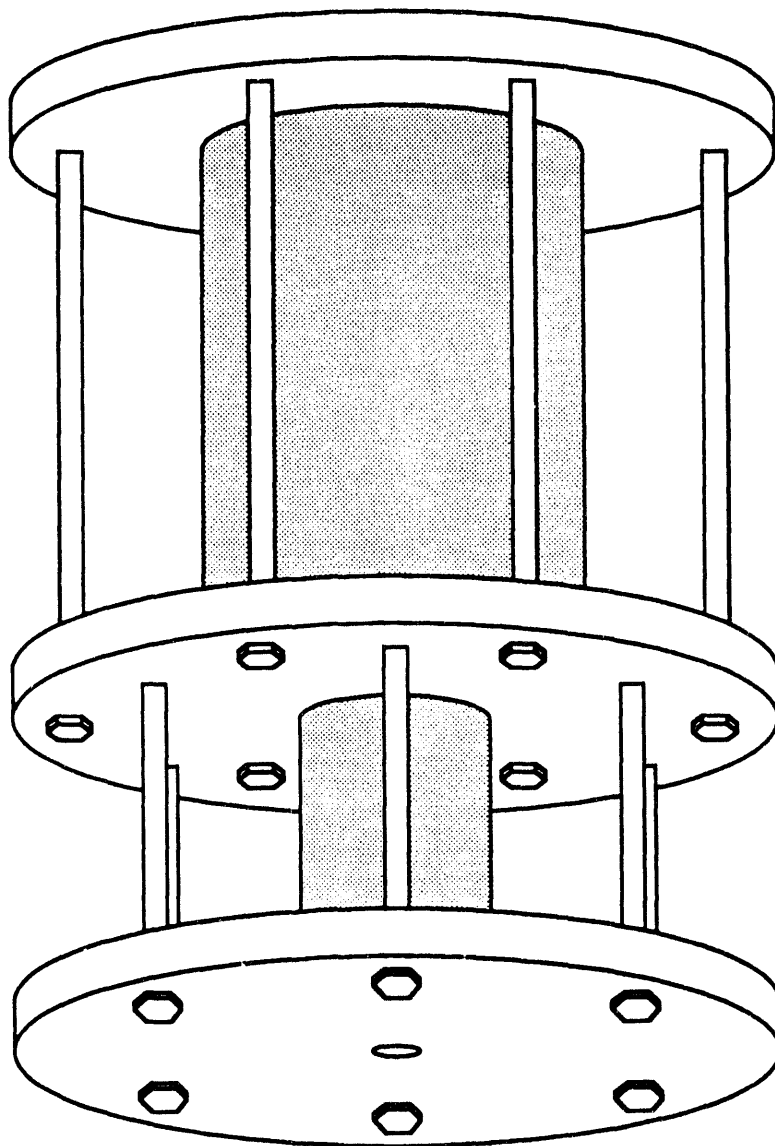


Figure 1

Reactor Assembly

Cut-in-Half View

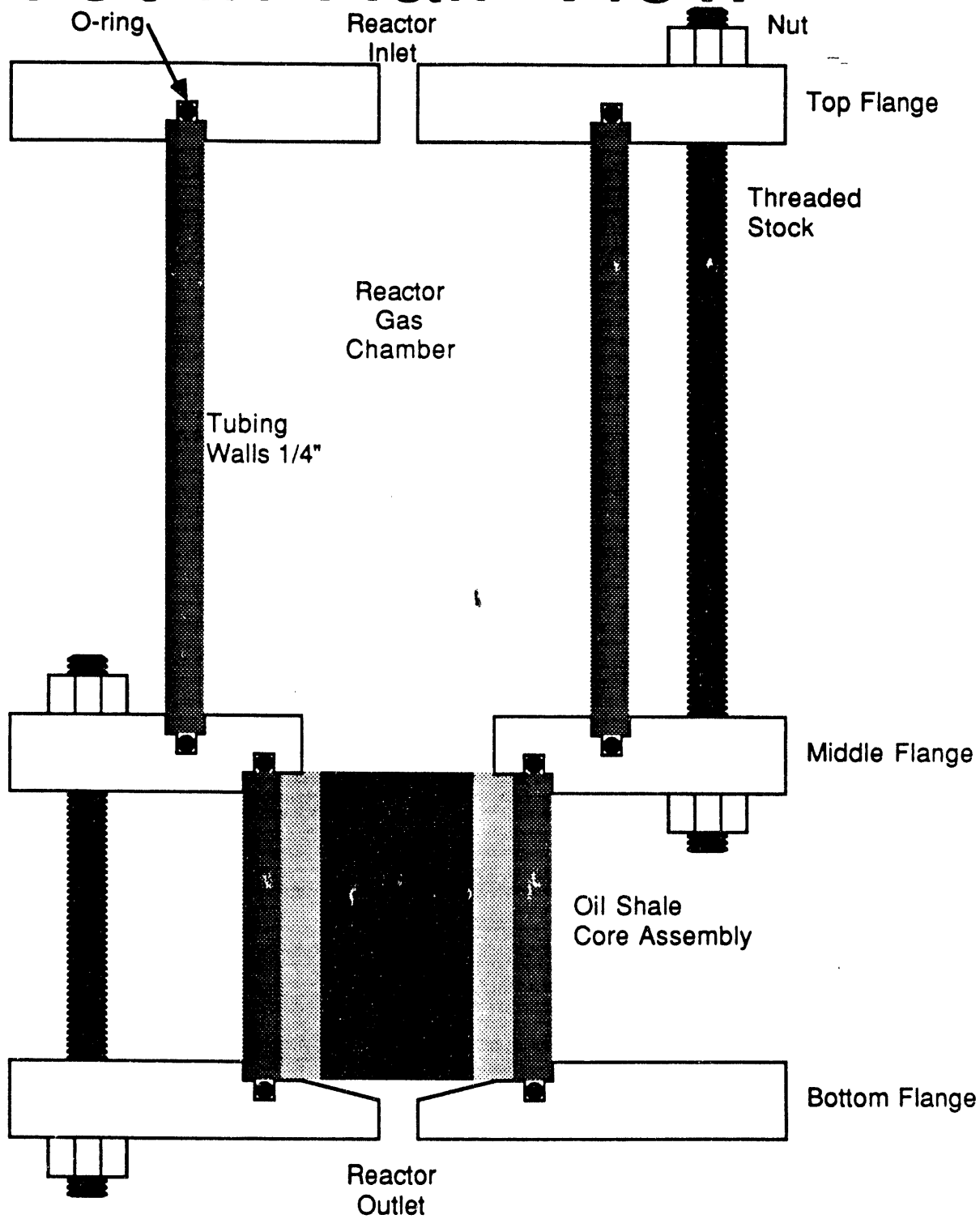


Figure 2

DATE

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