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Changing Scene Highlights III

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Iowa State University, Ames, Iowa 50011

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Foreword

The Ames Laboratory is a multiprogram, government-owned, contractor-operated laboratory of the United States Department of Energy. Iowa State University is the operating contractor through its Energy and Mineral Resources Research Institute (EMRRI), which also administers the Iowa Coal Project.

The Ames Laboratory's activities range from the most fundamental research in the physical sciences to applications-oriented programs in the energy and environmental sciences. Many of the Laboratory's scientific group leaders are also members of the University faculty, and a substantial part of the basic research is performed by University graduate students who hold research assistantships in the Laboratory.

Multi translates to interdisciplinary in many of the Laboratory's programs. Here interdisciplinary really means interpersonal because the Laboratory is an aggregate of research and support people who establish the identity of the Laboratory.

For this reason the emphasis in this biennial report is on the people performing the research. And the research reported covers broad topical areas rather than research along strict disciplinary lines. By extrapolating a little beyond the basic science reported we have attempted to show the integration of basic and applied programs in the Ames Laboratory's approach to energy research.

Robert S. Hansen
Director, Energy and Mineral
Resources Research Institute

CHANGING SCENE HIGHLIGHTS III

Ames Laboratory, DOE*, and
Energy and Mineral Resources
Research Institute
Iowa State University
Ames, Iowa 50011

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Hydrogen in Our Energy Future

The hydrogen atom has the most simple atomic structure in the universe; one proton and one electron. Thermonuclear fusion of hydrogen creates the power of the sun and is the first step in the formation of all the rest of the elements. Scientists from the United States and the Soviet Union are collaborating in a massive effort to replicate the solar furnace on earth so that it can be tapped as a heat source for the generation of electric power.

But even in the absence of fusion power devices, hydrogen gas will be a key element in America's fuel future. It might be used as a combustible fuel itself, and is a major aspect in the liquefaction or gasification of coal. The idea of burning hydrogen as a fuel is attractive. The product of its combustion is water—no hydrocarbons, carbon oxides, or sulfur compounds would be released into the air. Nitrogen oxides would be the only pollutants, as these compounds result from high temperature combustion of most fuels with air.

Hydrogen gas is highly volatile, but storage and handling are safer today than in the past, when disastrous fires like that of the Hindenburg caused the abandonment of the great hydrogen lighter-than-air ships. NASA uses hydrogen as a rocket fuel and has developed safe means for its transportation and storage. However, these systems are cumbersome and expensive. Scientists at Ames Laboratory are investigating the diffusion of hydrogen into metals and compounds. On the one hand, this may be the means for more effective storage of the gas, since some metals can absorb and store more hydrogen per unit volume than can a container of the same volume filled with liquid hydrogen. On the other hand, the ease with which hydrogen diffuses into metals can be a serious problem if a metal becomes brittle and fractures as a consequence. Ames Laboratory studies address both sides of the coin.

A number of novel compounds have been evaluated for their tendency to "soak up hydrogen." A compound's ability to readily store hydrogen appears to depend on the presence of conduction electrons it has available for bonding. Salts, which are insulators, do not have these conduction electrons, and will not absorb hydrogen. The group working with Dr. John D. Corbett, assistant program director, materials chemistry, has found that metallic halides, which are both metals and salts at the same time, will react with hydrogen. Of eight metallic halides studied so far, all have formed new hydride phases.

John D. Corbett

Nearly all of the compounds studied have been synthesized for the first time at the Ames Laboratory and were initially difficult to produce. As an example, zirconium monochloride was first produced by reacting a strip of zirconium with zirconium tetrachloride in a sealed tantalum tube for several weeks at 700 to 800°C. ZrCl crystals produced on the strip were removed and analyzed. Once the formula and some properties of the halide were known, a faster, higher yielding process was developed. Fifty grams of ZrCl are now produced in one or two weeks at 800-850°C. X-ray diffraction studies showed that the structure contains homoatomic layers sequenced Cl-Zr-Zr-Cl so as to generate a series of four-layer sheets. The layers are said to be "close-packed," similar to the way the layers in a crate of oranges would be—one layer of oranges nesting in the indentations formed by the layer below, (see Figure 1) only here layers of different size occur in pairs, corresponding to zirconium and chlorine atoms.

The four layer sheet structure of ZrCl is without precedent. Each zirconium atom has three metal neighbors in the next layer at 3.09 Å (an angstrom, Å, is 10^{-8}cm), six metal neighbors in the same layer at 3.42 Å, and three chlorine neighbors in the opposite layer at 2.63 Å. The strong metal-metal bonding is the most significant bonding feature of this structure, as

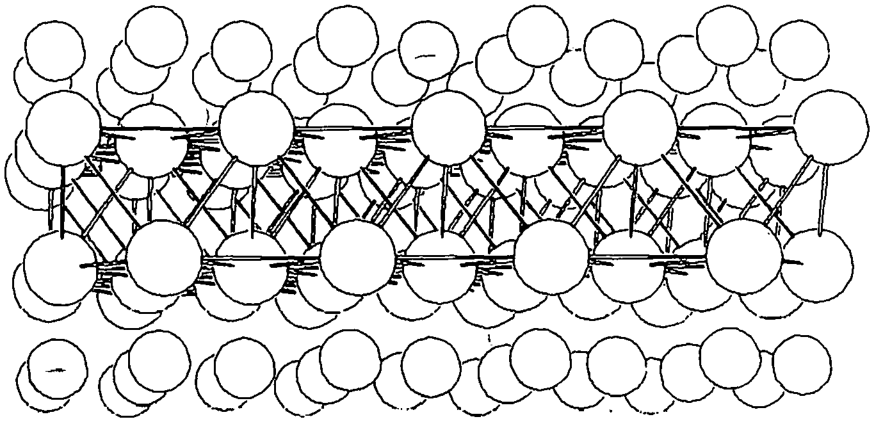


Figure 1. The layered Cl-Zr-Zr-Cl sequence. The small atoms are chlorine and the large atoms are zirconium.

revealed by the short distance between zirconium layers, 3.09 Å, which compares with 3.19 Å for 12 nearest neighbors in zirconium metal. This layered structure classifies ZrCl as a two-dimensional metal, its metal atoms being arranged in a two-dimensional plane rather than in a three-dimensional lattice. This is consistent with the graphitic character of the compound and its tendency to form polycrystalline structures and to flake off in sheets.

ZrCl and similar structures such as zirconium bromide (ZrBr) have been shown to readily accept and release hydrogen. The initial prediction was that relative hydride stability is largely determined by the competition between the metal cores and the hydrogen for conduction electrons, and that a partial replacement of conduction electrons by a more stable anion such as halide (Cl or Br) should lead to an increase in hydrogen dissociation pressure. This rather simple prediction has been borne out. It appears that the hydrogen readily passes into and out of the interstices between the zirconium layers. The ease with which these compounds react with hydrogen suggests potential catalytic applications such as in the rearrangement of hydrogen in small hydrocarbon molecules. This sort of rearrangement of hydrocarbons is the basis of such processes as catalytic cracking in the petroleum industry and in the liquefaction or gasification of coal. (See the accompanying story on catalyst research.) The catalytic possibility was an unexpected benefit of the hydrogen storage research.

This work has led to a great deal of interest in other so-called lower dimensional materials. While ZrCl forms two-dimensional layers, $ZrCl_3$ forms one-dimensional infinite chains of Zr atoms contained in hexagonal close-packed chlorine layers (see Figure 2). Strong bonding only in the metal chains makes the material exceedingly fibrous. Zirconium forms similar one-dimensional compounds with the other halides, bromine and iodine. Although very good directional electrical conductivity, possibly superconduction might be anticipated with these structures, initial tests have not revealed either behavior. Further work with the compounds has disclosed that they can be seriously nonstoichiometric, i.e. do not rigorously maintain the numerical relationship of one Zr atom to three halogen atoms. This implies that vacancies may occur in the chains upon oxidation, as well as displacement of zirconium ions into the otherwise vacant channels in the chlorine structure either spontaneously or upon reduction. This may provide a clue to the failure to detect the anticipated electrical conductivity and poses the possibility of investigating metal-metal chains of varying length, where macroscopic conductivity measurements would be useless.

Ken Poeppelmeier, graduate student with Corbett's group, recently synthesized totally new types of one-dimensional metallic structures that occur in a most unexpected place, the lower scandium chlorides. Heptascandium decachloride (Sc_7Cl_{10}) is formed of infinite chains of scandium, actually constructed from two parallel chains of scandium octahedra which are extremely distorted and share a common edge, while pentoscandium octachloride (Sc_5Cl_8) contains chains of single octahedra (see Figure 3). Both scandium chains are surrounded by a sheath of chloride atoms that also bridge to and between isolated Sc(3+) metal ions located between the chains. The shortest Sc-Sc distances in the two structures, 3.15 Å and 3.02 Å respectively, are remarkable in comparison with 3.28 Å in scandium metal. These strongly bonded and well-separated metal chains and the resulting physical properties suggest that this compound also can be described as a crystalline analogue to Dr. John Verhoeven's metal fiber composites (see accompanying story on extreme materials).

An important feature of Corbett's investigations is metal-rich compounds that exhibit extensive metal-metal bonding in their structures. The importance of metal-rich compounds is also a major characteristic of Dr. Hugo Franzen's study of materials stable at extreme tem-

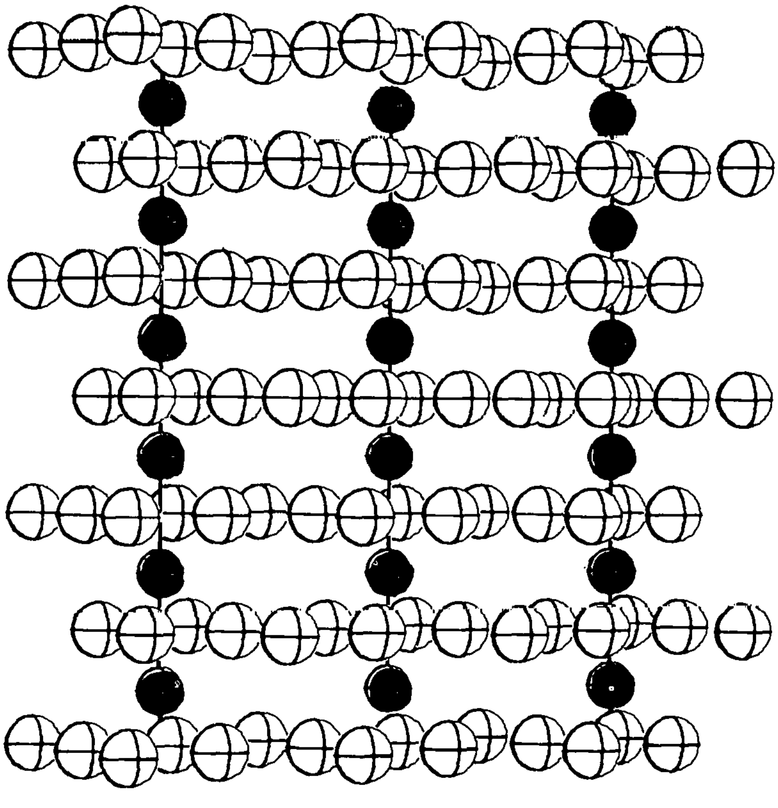
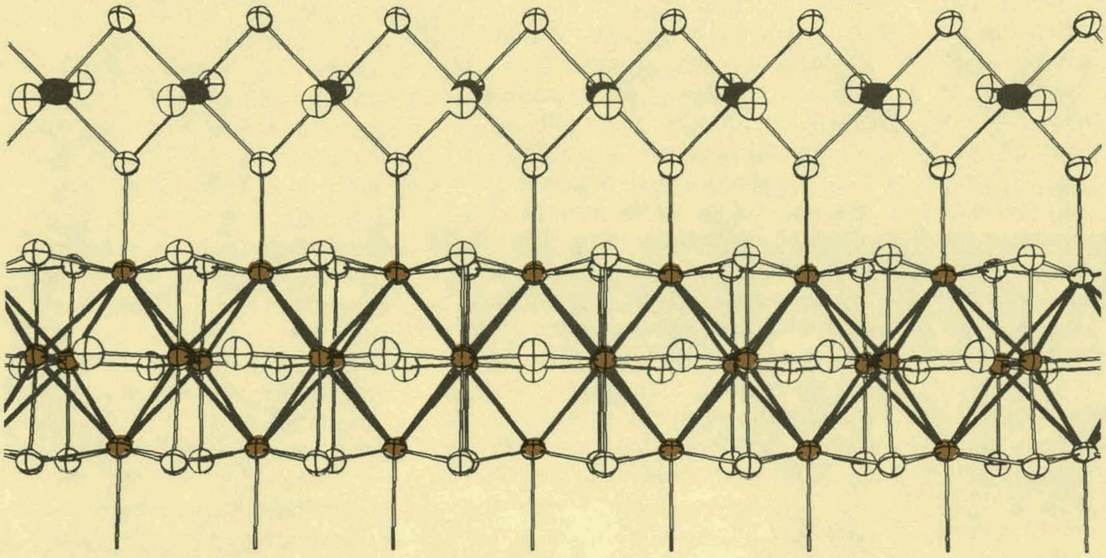


Figure 2. $ZrCl_3$, with proposed one-dimensional metal chains.



Sc_5Cl_8

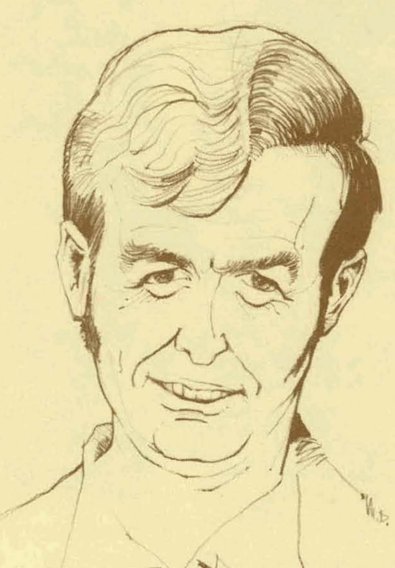
Figure 3. Scandium chains surrounded by a sheath of chloride atoms.

peratures. Likewise, metal-metal bonding is the key to Dr. Robert McCarley's research on compounds of the heavy transition metals. (See the accompanying stories on extreme materials and on catalysis.) Novel materials such as these have been the mainstay of the Ames Laboratory since its founding, and there is considerable cross-fertilization of knowledge from one group of researchers to another. Each group studying a particular class of compounds or particular aspects of chemical behavior relies upon the instrumentation and analytic capabilities of other groups in the Laboratory. This pooling of efforts is necessary to establish thorough characterizations of the materials under study. For example, some of the compounds synthesized by Corbett's group are subjected to x-ray and neutron crystallographic analysis by Dr. Robert A. Jacobson's chemical structures group, to nuclear magnetic resonance (NMR) studies by the researchers working with Dr. Richard G. Barnes, senior physicist, to secondary ion mass spectrometry (SIMS) by Dr. Gordon C. Danielson, senior physicist, and his associates in the Surface Analysis Laboratory, and to theoretical interpretation by Drs. Samuel H. Liu, section chief, theoretical research, and Bruce N. Harmon, associate physicist.

The creative exchange among these various groups is enhanced by the fact that scientists on the Ames Laboratory staff are also members of the Iowa State teaching faculty, and many of the research assistants are graduate students. Science is a learning process, and the need to answer questions, perhaps impertinently posed by students, can often lead to seeing problems in a new light, with subsequent insight. This creative interaction is important for building an understanding of completely new chemical structures in pursuit of the ability to tailor bonding arrangements in synthetic compounds and thus engineer specific properties.

The capabilities for x-ray diffraction characterization of the structures under investigation at Ames Laboratory have been tremendously expanded through the application of computer control and data processing by Jacobson and his coworkers. The system, developed over the last six years, can now do in two days what once required a month. Before the computer technique was perfected, crystallographic studies required time consuming film techniques to determine the orientation and size of the unit cell, the repeating unit in a crystal, and to record the diffraction intensities which carry structural information. The sample would have to be positioned in various orientations and for each of these the resulting diffraction patterns would have to be recorded on film and the intensities measured. Exposure times of 1½ to 2 hours for each film were typical.

Now with direct computer control of the instrument, the computer oscillates the crystal so that approximately one minute exposures are made on the film. The positions of about a dozen strong diffraction maxima occurring on these pictures are given to the computer. The com-

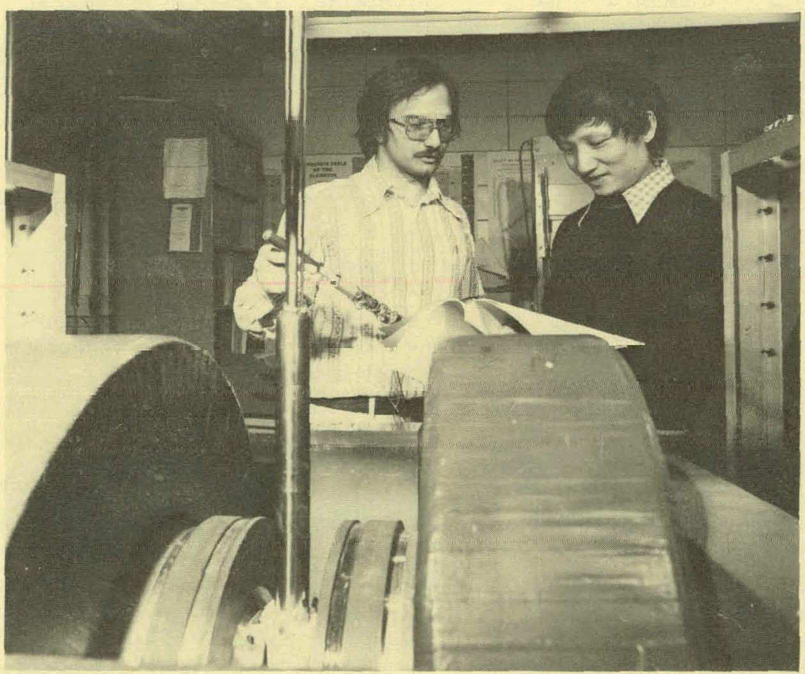


puter then determines the size and orientation of the unit cell and assigns crystallographic indices—numerical codes—to the diffraction maxima, after which the instrument automatically measures all the intensities.

The computer currently in use can simultaneously control three diffractometers while doing background calculations in a time-sharing mode and controlling another special experimental measurement series on the side. X-ray diffraction is useful for the structure studies of these novel compounds, but determining where the absorbed hydrogen resides in the structure and identifying the atomic environment at those sites requires neutron diffraction. The tiny hydrogen proton is a poor x-ray scatterer, but neutrons radiated on a crystalline solid will briefly form a compound nucleus with hydrogen or other materials in the solid. Neutrons, when re-emitted from each nucleus of the crystalline solid, interfere with each other to form a diffraction pattern which can be analyzed to determine the composition of the material.

While neutron diffraction averages the position of hydrogen in the structure, Barnes and his coworkers use nuclear magnetic resonance (NMR) to determine the location of hydrogen within the structure. Hydrogen nuclei behave like tiny bar magnets and will line up when placed in a strong d.c. magnetic field. The direction of alignment of the nuclei may be altered by radio frequency electromagnetic radiation if the axis of the RF coil is at a right angle to the magnetic field. The frequency at which energy is absorbed, changing the direction of nuclear alignment, is known as the resonant frequency, and for the hydrogen nucleus in a magnetic field of 14,000 gauss is 60 MHz. The resonant frequencies for identical nuclei in a constant external field vary slightly with the chemical environment of the nucleus (about 1 to 10 ppm for hydrogen). By varying the RF radiation over a small range, e.g., 1000 Hz, an absorption spectrum is obtained from which information about the structure of the material and position of the hydrogen can be deduced. (For a discussion of techniques necessary in applying this method to solids, see the discussion of the application of NMR to structure studies of coal on page 50.)

Richard Barnes



Phil Hornung, graduate assistant, and Tzong Hwang, postdoc, check probe which contains sample for pulsed nuclear magnetic resonance (NMR) tests for hydrogen diffusion in metals. Cylinders in foreground form an electromagnet.

To develop an understanding of how hydrogen is stored in some of the metals produced at the Ames Laboratory, Danielson, Dr. Albert Bevolo, associate physicist, and Howard Shanks, physicist, are depth profiling materials to determine whether hydrogen storage is a surface effect, or if hydrogen penetrates farther into the material. The profiling will indicate how much hydrogen can be stored by a given material, and will help determine which materials can store the most hydrogen in the smallest weight and volume.

The profiling apparatus is called a secondary ion mass spectrometer (SIMS) which detects hydrogen nuclei (protons) that x-ray detection methods cannot see. A secondary ion is one that is ejected from the material by an incident ion bombarding the surface. The secondary ion is detected, and its energy measured by the mass spectrometry equipment. Each impurity present in a material emits ions having energy levels characteristic to that impurity. Each energy level is recorded, and the impurities present are determined by comparison with known data.

Gordon Danielson

Albert Bevolo

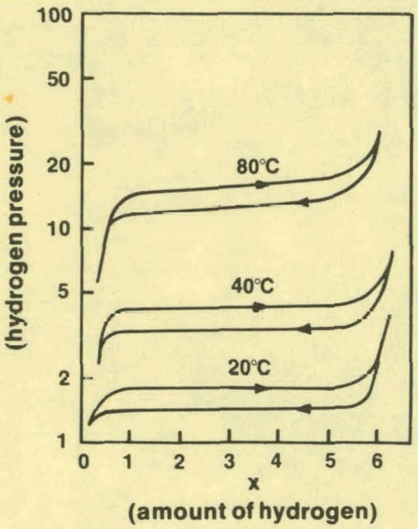
Howard R Shanks

Gordon Danielson (standing) and Albert Bevolo discuss secondary ion mass spectrometer (SIMS) test results.

Karl Gschneidner, Jr.

Dr. Karl Gschneidner, assistant program director, metallurgy and ceramics, has discovered that $LaNi_5$ is one of the more reversible hydrogen pick-up materials. Hydrogen stored in $LaNi_5$ can be recovered simply by placing the material in a vacuum.

The lanthanum-nickel compound is another material that can gain a greater concentration of hydrogen per volume than is possible by hydrogen alone in a liquid state. It will pick up seven hydrogen atoms to form the compound $LaNi_5H_7$.



$LaNi_5H_x$: pressure-composition isotherms measured in absorption (→) and in desorption (←).

(Data from F. A. Kuijpers and H. H. van Mal; a slight correction was applied for the absorption capacity.)

Lanthanum and nickel are fairly abundant elements, so cost, not availability, could be a problem with using them for large-scale hydrogen storage. The compound is also too heavy for most transportation fuel storage applications.

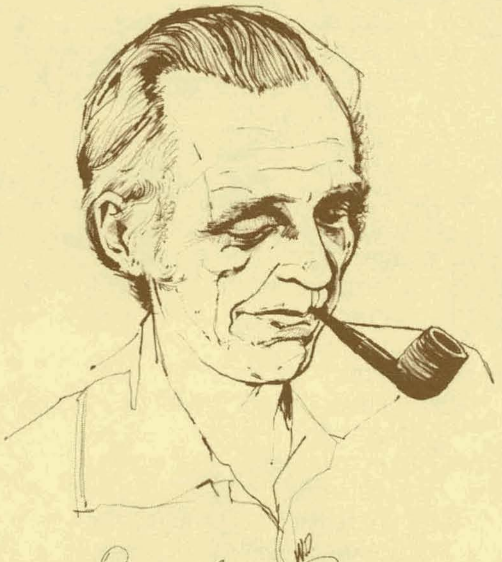
Temperatures are being varied to find the best condition for hydrogen uptake by $LaNi_5$. At very low temperatures, the electronic and vibrational characteristics of the compound when it contains hydrogen can be examined. The goal is to find a relationship between these characteristics and the amount of hydrogen that $LaNi_5$ or other compounds will absorb. From determined relations it may be possible to predict other compounds that will be good hydrogen storage materials. Promising compounds produced are supplied to other Ames Laboratory researchers who analyze their internal structure.

Many of the compounds surveyed have a serious weight disadvantage as storage systems for hydrogen, and a great deal of experience will be necessary before the promising ones admit to easy synthesis. While this work continues, the other side of the coin, metal deterioration, is also an active study. Metal embrittlement is a problem for gaseous hydrogen storage containers and transport systems, for coal gasification and liquefaction, and for solar conversion systems, as well as for fission and fusion nuclear systems.

The only way to eliminate the deleterious effects of hydrogen on metal is to prevent the hydrogen from contacting the metal. Since this is impossible for most energy systems, it is necessary to seek a solution by understanding the mechanism of this effect, and, by studying the influence of composition and structure on the process.

Metal embrittlement is one of the major interests of Dr. Tom Scott, section chief, mechanical properties, and his associates. Special emphasis has been placed on the embrittlement mechanism of vanadium, niobium, and tantalum because these are likely candidates for cladding materials in reactor core tubes containing radioactive fuel and for containing the reaction zone of a fusion reactor. At elevated temperatures these metals will soak up considerable amounts of hydrogen without destroying the cohesion of the metal lattice. This can create a serious problem if the metal with its "solution" of hydrogen subsequently cools down below a critical temperature. The composition may undergo a phase transition in which a new structure arises; for example the hydrogen may "come out of solution" and form a hydride of the metal. The phase transition may be accompanied by fracturing or even catastrophic collapse of the metal. There is considerable debate over the exact sequence of events in such failures. The formation of the hydride may be the key mechanism, or local de-cohesion of the metal lattice by the hydrogen may induce fractures. Further work is necessary to answer these questions. And these are not just academic questions. Adequate answers are necessary if accurate predictions of the behavior of the metal are to be made, such as whether or not the lining of a nuclear power reactor vessel would survive the stresses induced by sudden cooling consequent to an emergency shutdown.

Tom E. Scott



David T. Peterson

The role of interstitial impurities in these degradation processes is also poorly understood and is thus an important focus of attention. Work by Dr. David Peterson, senior metallurgist, has led to a method for extracting the interstitial impurities—carbon, oxygen, and nitrogen—from vanadium. A layer of titanium, condensed on the outside of the specimen, reacts with the impurities as they diffuse to the layer (see Figure 4). The inward diffusion of titanium is only one-millionth as fast as the diffusion of the impurities through the lattice of the vanadium, so only a thin layer of the specimen is alloyed with titanium and this can be removed by electropolishing. The resistance ratio of vanadium has been increased from 50 to more than 700 by this method. The interstitial concentrations have been reduced to: oxygen—6 ppm, carbon—2 ppm and nitrogen—1 ppm. This "external gettering" process is being tested on other metals whose properties are affected by interstitials.

Techniques have been developed to study the movement of hydrogen and deuterium (one proton and one neutron) atoms in the metals. Diffusion coefficients can be established for the migration of the "dissolved" H and D in the group V metals (V, Nb, Ta). Such migration has been investigated as a consequence of pressure differentials (squeezing the atoms out of one end of a sample, forcing them into the other), thermotransport (differential heating), and electrotransport (the H and D atoms are induced to move by an applied electric current). These studies are being extended to investigate the effect of "trapping" of the H or D by both interstitial impurities and by metallic solutes with a high affinity for hydrogen. Trapping by oxygen in vanadium and niobium has been reported to greatly modify diffusion behavior. The thermotransport results should be helpful in understanding and controlling the interactive effect of hydrogen and the other interstitial solutes on embrittlement of these cubic metals at low temperatures. Thermotransport is an important factor when hydrogen or deuterium are present in a material that is subjected to a temperature gradient, such as will exist in most energy conversion devices.

Control over the presence, possibly even the addition to the material, of a solute that traps the hydrogen or deuterium would make it theoretically possible to design alloys in which there would be no tendency for them to migrate to either the higher or lower temperature regions. The "gettering" effect will be an important research tool in establishing this sort of fine control over the "impurity" content of these metals. Nuclear magnetic resonance studies by Barnes and his co-workers are especially useful in these investigations because the deuteron possesses an electric quadrupole moment; thus, its NMR is sensitive to crystalline symmetry and structural instabilities. This provides a powerful means for determining details of structural changes at the level of single atoms.

Laboratory researchers intend to extend this group of studies to the consideration of tritium (a proton and two neutrons) when techniques to handle the radioactivity of the tritium in such tests are devised. The study of electrotransport will also be extended to other metals such as beta-titanium alloys and the alkaline earth metals to test theories and models for hydrogen in metals over a wider range of matrix properties.

Hydrogen attack is a phenomenon of direct concern in coal gasification and liquefaction—processes in which hydrogen gas will contact materials, especially ferrous metals, at elevated temperatures and pressures. Scott is engaged in a study of the effects on steel of an environment that simulates a "coal dissolver." Various coal liquefaction methods, whether the Synthoil process being developed at the Pittsburgh Energy Research Center (PERC) in Pennsylvania, or the solvent-refined coal (SRC) process pursued by PAMCO (a Gulf Oil subsidiary), use a high pressure, high temperature reaction of coal with hydrogen gas to turn the coal into a liquid or semi-

Figure 4

solid that has most of the sulfur and ash constituents removed. The Synthoil process uses a coal "slurry," a mixture of crushed coal, oil (produced by the process, reintroduced into the system as a solvent to facilitate the reaction), and hydrogen gas (introduced into the process). The reaction vessels or "dissolvers" and the tubing used to preheat the coal slurry are subjected to temperatures averaging 850°F (range 470° to 1600°F), and hydrogen partial pressures of 2000 to 4000 psig, in combination with a variety of noxious gases, especially H_2S from the hydrogenation of sulfur.

Because liquefaction operations are in their infancy, there is little information available to indicate how well the construction materials for these vessels will withstand the stress. Frequently it has been claimed that experience from the petrochemical industry can be applied to coal liquefaction processes. However, the severe combination of temperature, pressure, and particularly noxious gases involved in liquefaction is not present in petrochemical processes. The integrity of liquefaction vessel materials must be evaluated under the conditions of their application.

Scott, along with Charles Owen, associate metallurgist, Lester Reed, senior research technician, Charles Wood, graduate student, and Dr. Shen-ann Shei, postdoctoral fellow, is engaged in testing 2¼ Cr-1 Mo steel under conditions that simulate the environment of the dissolver. After extensive tests to establish baseline data, steel bars, some notched to induce hydrogen concentration at the stress points replicating fracture processes, will be placed under tensile stress and subjected to temperatures up to 900°F and pressures up to 4000 psig, in vessels containing actual coal slurry obtained from PERC. This project has necessitated the construction of special pressure vessels and safety test facilities located away from the main Iowa State campus. The experience gained should answer the immediate practical question of suitable materials and safety standards for the pilot plant of PERC's Synthoil project, as well as extend our knowledge of hydrogen embrittlement in steel.

Catalysis—Making Chemical Reactions Work

A catalyst is a substance that increases the rate at which a chemical reaction occurs, without itself becoming permanently involved in the reaction or its products. Knowledge and application of catalysts make possible the multitude of modern chemical industries, ranging from the hydrogenation of animal and vegetable fats into edible margarine by a nickel catalyst, to the catalytic cracking of crude petroleum, forming useful hydrocarbons as different in character as paraffin and high octane gasoline. Nearly one-fifth of all the products manufactured in America require a catalyst somewhere in the production cycle. Life itself is based on systems of catalysis, homeostasis being the result of control processes by enzymes—biological catalysts.

While catalysts make possible the activities of high technology, often they are needed to clean up harmful by-products of this technology. An example of the need for thorough understanding of the processes involved in catalytic activity is the recent introduction of "catalytic converters" to automobile engines. Most noble metals will catalyze the reduction of nitric oxide (NO, an undesirable gas) in the presence of hydrogen (H) and carbon monoxide (CO, available in auto exhaust), but produce various amounts of nitrous oxide (N_2O) and ammonia (NH_3) which are themselves undesirable. If platinum is used to catalyze the reaction, the product is mostly ammonia, but ruthenium (Ru) is unique in that it produces NH_3 in one temperature range, and mostly nitrogen gas (N_2) in another range. The N_2 is certainly preferred, inasmuch as N_2 makes up about 80 percent of the air we breathe.

Dr. Robert S. Hansen, director of the Ames Laboratory, and Thomas W. Orent, a graduate student in the surface chemistry program of the Laboratory, have developed an explanation for the two different Ru reactions. This understanding is necessary to the sound application of catalytic reactions, and at the same time advances our ability to engineer desirable catalytic effects in a fundamental sense. The reaction for reducing the NO to the desired N_2 involves *heterogeneous* catalysis; *i.e.*, the catalyst is in a solid state, while the reactants are in a gaseous state. The term heterogeneous merely denotes this difference in physical states. (If the catalyst were in the same state as the reactants, *e.g.* liquid in a reaction between liquids, the catalysis would be called *homogeneous.*)

Since the ruthenium is a solid, the catalysis occurs at the surface of the metal. The key to the action of the catalyst is the metal's ability to temporarily trap the reactants at its surface by a process called adsorption. Adsorption means that the substance which has been captured stays on the surface, as distinguished from absorption, which denotes a process of one substance penetrating into the other.

Adsorption occurs because of the unique electrical state of the surface layer of atoms in the metal. Any atom on the surface is surrounded by similar atoms in the same plane or in the plane below, to which it is attracted, but there are none above it. Because of this, solids possess a surface energy akin to, but much stronger than, the surface tension of a liquid.

Robert S. Hansen

Each surface atom has its valency requirements unfulfilled. The force which drives molecules to react with and be adsorbed by solid surfaces is the tendency of this free valency, either covalent or electrostatic, to become saturated. This kind of adsorption, in which new chemical bonds are formed, is generally called *chemisorption*, to distinguish it from a weaker form of adsorption called *physical adsorption*, in which the forces attracting the molecules to the surface are of the same kind as those which hold molecules together in a liquid—van der Waals forces. (Van der Waals forces arise as a fluctuating electric moment in one molecule induces an electric moment in another molecule, and the two moments then interact.)

In somewhat simplistic terms, the heterogeneous catalyst acts by increasing the likelihood that the chemical bonds of the substances in a reaction will be changed by initiating that change, perhaps only slightly, as it attracts one or more of the reactants to its surface. In the case of nitric oxide and ruthenium, the nitrogen and the oxygen separate when the NO molecule attaches to the surface of the Ru, and become independent atoms.

Orent and Hansen have done an extensive study of the behavior of N and O adsorbed on the surface of Ru, and found that the key to the difference between forming desirable N_2 gas, as opposed to undesirable ammonia, is the formation of an oxide that actually disrupts the surface of the metal. They combined data from two separate methods for analyzing the activity at the surface of the Ru crystal with findings from numerous related studies to develop their solution to the puzzle. Low energy electron diffraction (LEED) pictures gave them hints of the physical arrangement of the surface and the atoms adsorbed onto it. LEED is a technique in which electrons of uniform energy in the range of 5 to 500 electron volts (ev) are bounced from the surface of the sample, and those electrons which have lost no energy are selected and accelerated to a fluorescent screen where the diffraction pattern from the surface can be observed. Auger electron spectroscopy (AES) was used to find out which atoms were present and giving rise to the surface pattern. An Auger electron is an electron spontaneously emitted from an excited atom; the excitation responsible for the emissions may be produced by x-ray illumination or by electron bombardment. The energy of the Auger electrons can be precisely measured and used to determine the species of atoms from which they were released. LEED and AES make very good complementary methods. In fact, for their investigation, Orent and Hansen used the same electron gun to bounce out the electrons for both the measurement and the pictures. Figures 1a and 1b are examples of the images obtained from the LEED.

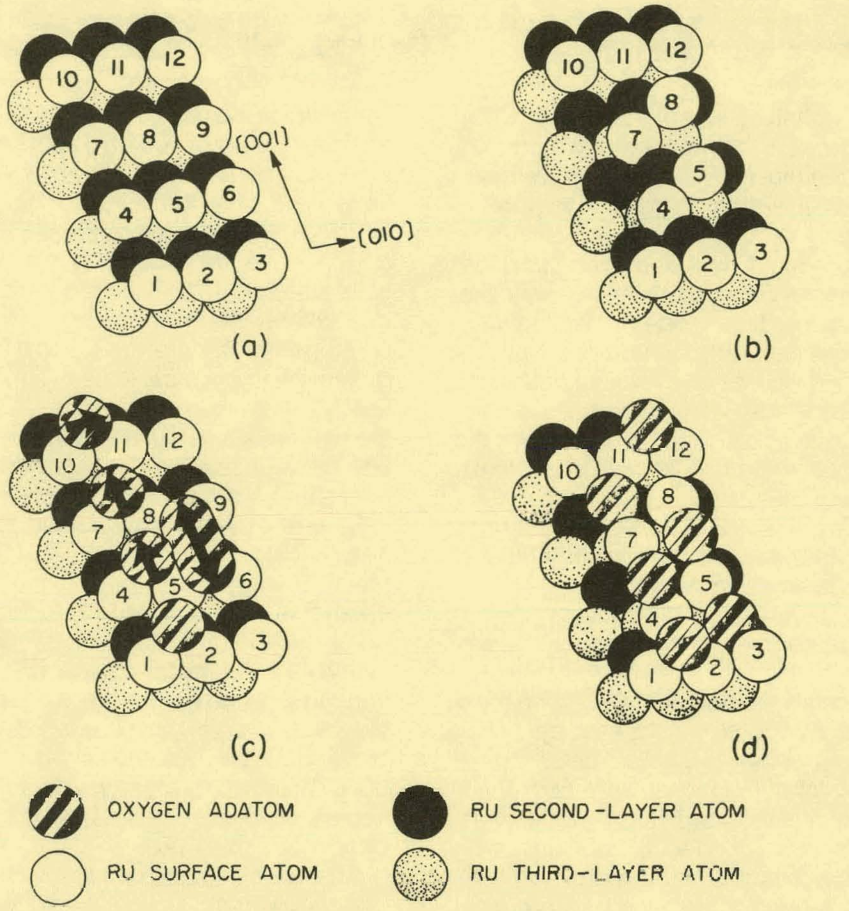


Figure 2. Oxide overlayer formed by the reconstruction of a Ru surface; (a) unaffected metal, (b) the reconstruction, (c) and (d) the oxygen adatoms in the model.

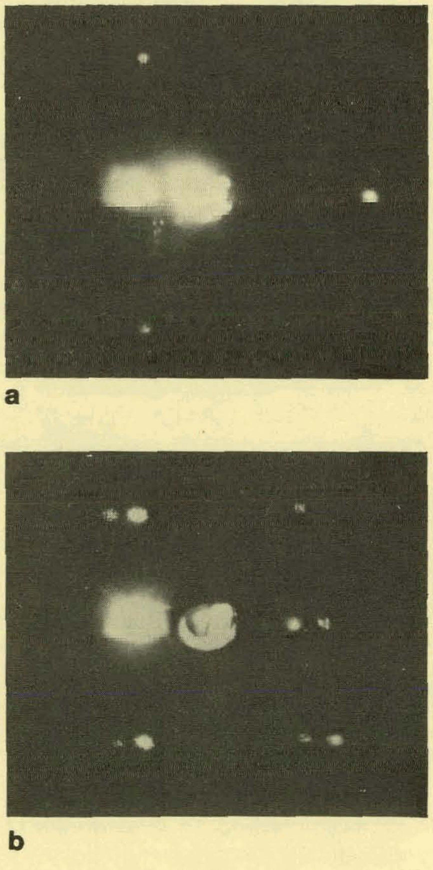


Figure 1. Examples of LEED images obtained from Orent and Hansen's study; (a) result from either NO or O_2 adsorbed on ruthenium at temperatures below 200°C, (b) result from either NO or O_2 adsorbed on ruthenium at 950°C.

It had been noted by other researchers that Ru, when slightly oxidized, was active in decomposing ammonia and forming hydrocarbons from carbon monoxide and H_2 gas, but that Ru in a reduced state did neither. Both forms equally reduced NO to N_2, but no reason for the NH_3 and CO immunity to the reduced catalyst was evident. Orent and Hansen found that at most temperatures nitric oxide (NO) was split upon adsorption and that N quite readily desorbed. The O however, formed different configurations depending upon the temperature. Figure 2 represents their explanation of the difference. At prolonged exposures to temperatures above 400°C the surface of the metal is reconstructed by the activity of the oxygen. Figure 2a shows the unaffected metal. 2b shows the reconstruction. The structure is formed by shifting the surface atoms in the two center rows from the four-fold holes formed by the underlying metal atoms to the adjacent three-fold holes which are only 1.6Å away. This reconstruction is accomplished without any change in the density of the surface atoms. Figures 2c and 2d show the oxygen adatoms in the model, 2c showing the necessary movement of the oxygen to give rise to the reconstruction. The geometrical configuration and electronic structure of the reconstructed oxide layer shown in 2d are different from those of the bare metal, and these differences change the catalytic activity. Since some of the oxygen adatoms are incorporated in the reconstructed metal, chemisorption may take place on either the metal atoms or the oxygen atoms.

Robert J. Angelici

A knowledge of catalysis may also give us a fuel alternative to gasoline. Chemical reactions of carbon monoxide with water which are seemingly very simple, can form a number of fuels: methanol, various other hydrocarbons, and even hydrogen gas. When represented theoretically the reaction is very straightforward. The hydrogen producing version is called the water-gas shift reaction:

$$CO + H_2O \rightarrow CO_2 + H_2.$$

Additional steps give rise to various hydrocarbons, e.g.

$CO + 2H_2 \rightarrow CH_3OH$ methanol
$2CO + 2H_2 \rightarrow CH_4 + CO_2$

methane and carbon dioxide, etc. The problem is that these reactions occur only at high temperatures and pressures, and frequently give a mixture of products. Finding the right catalyst might increase our ability to perform these reactions, however, and Dr. Robert J. Angelici is engaged in this search as a part of the Laboratory's Basic Energy Sciences program.

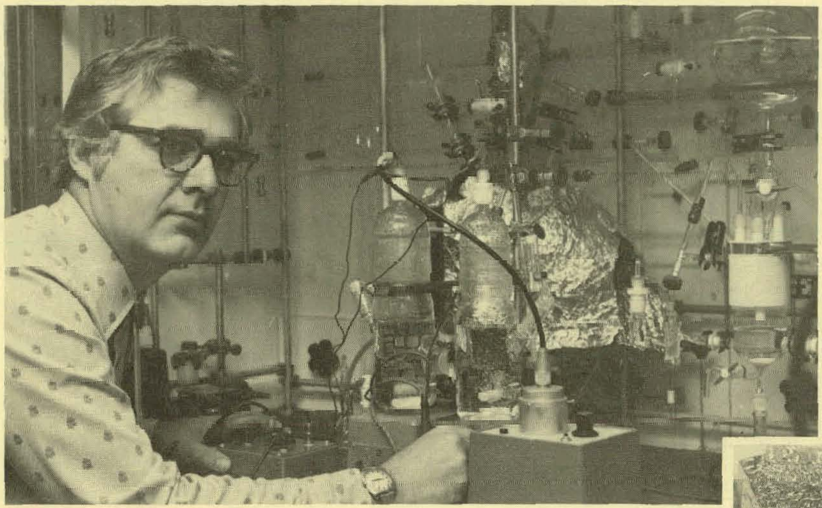
Until recently only heterogeneous catalysts have been reported to catalyze the water-gas shift reaction. However, one study has been reported in which $Ru_3(CO)_{12}$ was used to homogeneously catalyze this reaction in an organic solvent under mild conditions (100°C and 1 atmosphere CO pressure). Angelici's studies have shown that several other metal compounds can be used for the reaction:

$Mn_2(CO)_{10}$
$Co_2(CO)_8$
$(Rh(CO)_2Cl)_2$.

This suggests that one of these catalysts may allow the large scale preparation of H_2 under mild conditions that would avoid the costly equipment required by the high temperature, high pressure facilities required by a number of proposed Department of Energy techniques. These involve heterogeneous catalysts that are poisoned by sulfur. Sulfur poisoning is an important problem, since many of the processes under study involve coal, and sulfur is a major contaminant in coal. For organic reactions, homogeneous catalysts usually give greater product selectivity and proceed under milder conditions. Angelici's work uses dimethoxyethane solvent to coax the process along, and preliminary studies have shown that the addition of amines or potassium hydroxide (KOH) as a co-catalyst significantly improves the yield of H_2.

Ruthenium thin films are being used by Michael Slaughter, a graduate student with Hansen's catalytic group, to study the methanation reaction ($CO+3H_2 \rightleftharpoons CH_4+H_2O$). A temperature and pressure region has been found in which only this simple reaction producing methane and water is believed to occur. By developing a mechanism for the methanation reaction on ruthenium, Slaughter and Hansen hope to be able to comment on the possible mechanistic interpretation of more complicated H_2/CO interactions (alcohol, aldehyde, ketone, paraffin, olefin formation). In this photo, a ruthenium film is being deposited by electron bombardment of a polycrystalline ruthenium disc.

Another possible method for producing hydrogen is by the electrolysis of water into oxygen and hydrogen gas. Here again, a good deal of energy is required to split the H_2O molecule into its constituent gases. The use of platinum electrodes considerably increases the rate of the reaction, so much so that the operation is actually called electrocatalysis. The problem with this method is the prohibitive cost of platinum. Substitutes have been proposed for the platinum, and one of the prime candidates has been sodium-tungsten bronze. Contradictory results for this substitution have been bantered back and forth in the literature over the last ten years: a contention that the tungsten bronze was as good as platinum, a counter claim that platinum contamination had led to erroneous results, the subsequent finding of no



Howard Shanks is shown here with the Ames Laboratory-developed fuel cell which incorporates platinum-doped sodium-tungsten bronze as an electrocatalyst.

Crystals of sodium tungsten bronzes, $Na_x WO_3$.

Robert E. McCarley

catalytic effects even with massive platinum doping on the bronze, and so on back and forth.

Recent work by Howard R. Shanks, Drs. Albert J. Bevolo and Gordon C. Danielson, and graduate assistant, Michael Weber, all physicists in the new Surface Analysis Laboratory, has established that doping the sodium-tungsten bronze ($Na_{0.7}WO_3$) with a very small but precisely controlled amount of platinum can produce electrocatalytic activity nearly as well as platinum—at a fraction of the cost. The narrow range of platinum content and the incomplete characterizations of past studies evidently caused the contradictory results that had contributed to the decade-long dispute. Electrodes constructed with the composition established by the physicists should work equally well for producing H_2 by electrolysis of water, or for producing power by the oxidation of H_2 in a fuel cell. *Any catalyst that increases the rate of a reaction must also increase the rate of the reverse reaction, as the more complete definition of a catalyst is any substance that increases the rate at which a chemical reaction approaches equilibrium, without itself becoming permanently involved in the reaction.*

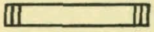
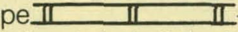
Most metal catalysts act in a heterogeneous manner; they are usually solids while the reactants are most often liquids or gases. As already noted in the discussion of the water-gas shift reaction, homogeneous catalysts usually have the advantage of working at milder conditions than heterogeneous ones and should have much higher efficiencies.

Dr. Robert McCarley, senior chemist, and his co-workers are engaged in synthesizing a class of materials which they anticipate will combine a metal-like surface activity with a catalyst that can work in a homogeneous mode. This work is a pioneering investigation of the relationship between molecular and electronic structure and the chemical and physical properties that these structures produce in compounds of the heavy transition metals containing a large proportion of metal-metal bonds.

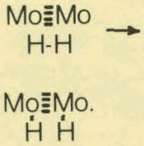
McCarley's intention is to develop the ability to understand and control synthesis of metal-metal bonds so as to generate compounds with desired properties whether in the simple case of two metal atoms joined to form a discrete molecule or ion, or in the much more complex cases where large clusters, extended chains, or two- and three-dimensional networks are desired. In this aspect his work is similar to the work of Drs. Hugo Franzen and John Corbett. (See the accompanying stories on extreme materials and on hydrogen storage and materials.)

A striking achievement of these investigations is the successful synthesis of a tetranuclear molybdenum halide anion, $Mo_4I_{11}^{2-}$. The key to this unique construction is the reaction of the carbonyl anion, $Mo(CO)_4I_3^-$, which releases carbon monoxide in a non-coordinating solvent so that the hypothetical intermediate MoI_3 has to react with itself in order to satisfy valence electron and coordination requirements, thus forming the metal cluster species.

The anion cluster could serve as the nucleus for the addition of other metal atoms so that still larger clusters may be formed with good control over the products. Further possibilities arise from inserting different metal atoms to form heteronuclear (mixed-metal) clusters.

The concept of using dimeric species with metal-metal triple or quadruple bonds in a controlled formation of "metal polymers" could lead to a rich variety of new materials, useful not simply for catalysis, but for an entire spectrum of as yet unimagined applications. McCarley has already worked with dimers of the type $Mo_2Cl_4(PR_3)_2L_2$ that can undergo condensation which results in the "addition" of two dimers containing Mo≡Mo bonds to form the rectangular cluster Mo_4 of the structure  having two Mo-Mo single bonds and two Mo-Mo triple bonds. This process could be repeated to form a ladder polymer of the type —a one-dimensional chain linked by strong Mo-Mo bonds.

Application of the Mo≡Mo complexes to catalytic hydrogen transport can be envisioned by the dissociation of an H_2 molecule:



$$\underset{\text{H-H}}{\text{Mo}\equiv\text{Mo}} \rightarrow \underset{\text{H}\quad\text{H}}{\text{Mo}\equiv\text{Mo}}.$$

This type of addition reaction to temporarily form the hydride might then provide the means for hydrogenation of complex hydrocarbon molecules, as in the breaking down of complex coal molecules to simpler structures. The metal-metal multiple bond in these complexes is very electron rich and should constitute a reactive center. Other molecules can be bonded in adjacent sites to facilitate the hydrogenation transfer. Such possibilities point to the use of these clusters either in homogeneous systems of catalysis at low temperatures or heterogeneous systems at high temperatures. The realization of these objectives require basic chemistry developments, including the control of synthesis of the metal-metal multiple bonds and an understanding of the factors that effect reactivity towards addition of hydrogen and small molecules.

Work on hydrogen storage in metals by John Corbett's group has led to catalytic possibilities similar to those of the metal cluster complexes. The experience with lower-dimensional metal compounds with high affinities for hydrogen such as the layer structure of zirconium bromide (ZrBr) indicates that these compounds may be effective hydrogenation catalysts. The examination of scandium halogen compounds that have extensive hydride forming capabilities suggest applications as isomerization catalysts. Results of this work show that Sc_2Cl_3 polymerizes hexene even more readily than ZrBr does. The creative interchange between these two groups of researchers continually sparks new insights into the amazing possibilities of these entirely new substances.

John O. Corbett

Materials—Expanding Their Upper and Lower Limits

Back in the early nineteenth century, when the French engineer Sadi Carnot first formulated a theoretical statement of the efficiency of heat engines, heat was still considered to be a subtle fluid and human understanding of the forces in nature had not yet been generalized into the unifying concept of energy. Now, in the last quarter of the twentieth century, the production and use of energy has become a critical social issue, but Carnot's statement, that the efficiency of any transformation of heat into work is dependent upon the differential between the temperature of the source of the heat and the temperature of its final destination at the completion of the cycle, still holds true. This means that the design of more efficient power generation systems will demand materials for their construction that can withstand higher temperatures. This is the case whether the heat for the system comes from fossil fuel or fission.

Most of Carnot's contemporaries believed electricity, as well as heat, to be a subtle fluid. The abandonment of that notion was a major step toward a theoretical synthesis of the general concept—energy. And just as we've abandoned the notion of electricity as a subtle fluid, we've also discovered that the key to efficiency with electricity is not high temperatures, as with heat, but rather extremely low temperatures. H. K. Onnes discovered in 1911 that when some substances are cooled to near absolute zero (or, toward the melting point of helium) the electrical resistivity vanishes. Such substances are called superconductors and are being developed for uses in the efficient transmission and storage of electrical power. Superconducting materials are also a key ingredient of the attempt to harness the power of the fusion reaction. The enormous explosive force of hydrogen fusion will require tremendous magnetic fields if these miniature suns are to be held in check; fields only made possible by the use of superconducting electromagnets.

Materials, then, are a key to future energy development—materials that can withstand very high temperatures and materials fabricated for use at very low temperatures. Both extremes are the subject of research at the Ames Laboratory. Since the Ames Laboratory was instituted at the end of World War II—indeed, even before that, back when the uranium for Fermi's pile at Chicago was extracted under the direction of Frank Spedding and Harley Wilhelm—Ames Laboratory scientists have specialized in the preparation of exotic and complex materials.

Separation techniques developed at Ames Lab have transformed the rare earths from their past behavior of clannish and reclusive hiding among each other into functional materials applied to everyday uses. Uses range from the creation of the vivid red in color televisions to a 30 percent increase in production capacity of petroleum refineries by catalytic application to the cracking processes. Building on the long tradition of innovations in materials research and development, Ames Laboratory scientists are engaged in formulating today's questions from what little was known yesterday about tomorrow's needs.

The establishment of fundamental knowledge on which to base the solution to technical problems is a creative process, and a major characteristic of creative processes is that results often violate expectations; that's what makes the knowledge fundamental. Such is the case with investigations by Dr. Hugo Franzen's high temperature chemistry group. Traditional valence theory would predict that it is impossible to form a compound from titanium and sulfur that contains two atoms of Ti to each atom of S. Working with a premise that compounds which are going to be stable at high temperatures may require high temperatures for their formation, Franzen succeeded in producing Ti_2S. The compound can be created by heating the titanium and sulfur reactants to extremely high temperatures in tungsten crucibles held in a vacuum. Contamination is avoided by driving the reaction with heat by induction from a coil connected to a powerful radio frequency electromagnetic generator.

Franzen's group is engaged in synthesizing and characterizing a broad range of such "metal-rich" compounds in order to determine

Metal-Rich Sulfides and Selenides Prepared in the Ames Laboratory

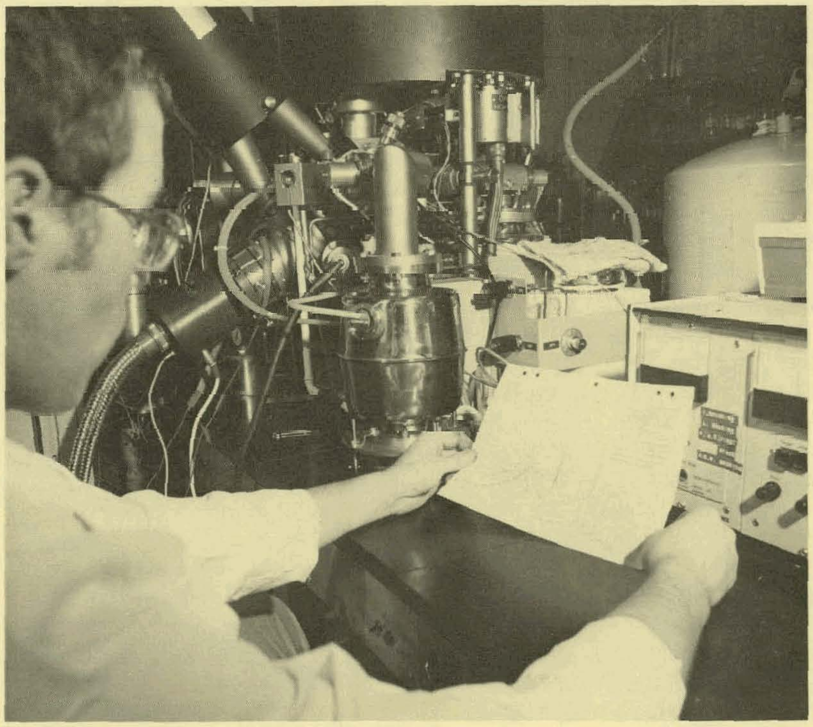
Ti_8S_3
Ti_2S
Zr_9S_2
Zr_2S
HfS
Hf_2S
$Nb_{21}S_8$
$Nb_{14}Sr$
Ta_6S
Ta_2S
Ti_2Se
Zr_2Se
Nb_2Se
Hf_2Se
Lu_3S_4

stoichiometries (the numerical relationships of components), structures, stabilities, and electronic behaviors in the 1500-2500K range. (To translate to Celsius, subtract 273°.) The compounds are analyzed by means of a wide variety of techniques: x-ray diffraction on solids (both crystals and powders), mass spectrometry of vaporized samples, and x-ray photoelectron spectroscopy. The photoelectron spectroscopy is possible only because the group is both a part of Ames Laboratory *and* a part of the chemistry department of Iowa State. The National Science Foundation provided the x-ray photoelectron spectrometer through an instrument grant to the chemistry department. Then, DOE contributed additional apparatus used in conjunction with the spectrometer.

The x-ray photoelectron spectrometer works by bombarding the sample with x-rays at known energies until electrons are bounced out of the sample. By measuring the kinetic energy of the liberated electrons, the binding energy can be calculated. Investigations of sulfides of Mg, Ca, Sr, Sn, Ti, V, and Mn indicate that the binding energy shifts of the sulfur inner core levels are different for the compounds in which a metallic d-band is present (the early transition metal monosulfides of Ti, V, and Mn) from those which have no d-band (the alkaline-earth monosulfides of Mg, Ca, and Sr). A band is a restricted range in which the energies of electrons in solids lie as understood in quantum-mechanical terms. The shifts in binding energy in the case of d-band interaction are about 1.5 electron-volts smaller than in the case of the classically ionic compounds. It appears that the d-band formation in the compounds with the transition metals increases the effective electronegativity of the metal relative to the sulfur, producing a covalency rather than an exclusively ionic bonding. This covalency effect stabilizes the compounds in structures which are unusually hard, metallic, and non-volatile at elevated temperatures. This is an important insight for the design and application of high temperature materials, not only for possible structural use, but especially for electrodes and protective coatings.

Photoelectron spectrometer results for the metal-rich compounds Ti_2S and Ti_2Se suggest that the interaction of the electrons in the p-orbitals of the chalcogens (S & Se) with the metallic d-band formed by the titanium is sufficient to reverse the electron transfer usually occurring in the majority of titanium chalcogenides. That is, these compounds appear to show a net transfer of electrons from the sulfur or selenium to the titanium. This opposes the traditional concept of electronegativity as an elemental property and suggests that electronegativity is more properly considered within the context of all the bonding interactions affecting a given element.

These investigations are being extended to the phosphides and aluminides as part of the effort to construct a model for the bonding in such compounds. The principal features of the model in these metal-rich compounds are the band which arises from metal-metal interactions, the interactions of the p-orbitals of the non-metal with the metallic band, and the apparent increase of the valence of the non-metal beyond that required to fill the valence shell. This model allows the prediction of properties in the transition-metal compounds such as increased stability from the effects of interaction by p-orbitals with the metallic band. Such a model should increase the ability of chemists to predict the thermal stability and electronic properties of materials which have yet to be prepared. Compounds formed with phosphorus have potential use in solar energy conversion and superconductivity. Highly stable aluminides could provide corrosion resistant coatings for high temperature applications.



James W. Anderegg, assistant chemist, examines a full-scale photoelectrometer scan to determine the elements present.


Doug Finnemore


John Verhoeven

While the high temperature chemists are interested in stability at high temperatures, Senior Metallurgist, Dr. John Verhoeven, and Senior Physicist, Dr. Douglas Finnemore, are interested in electroconductivity at very low temperatures. They are working on a process for producing very fine wires of niobium and tin in a copper matrix. It has been known for several years that Nb_3Sn is one of the best of all superconducting materials. Nb_3Sn has an unusually high critical temperature of 18.3K. The critical temperature is the temperature below which the electrical resistivity falls to zero: niobium alone is 9.09K and tin is 3.72K.

The development of practical high-field superconducting magnets may be said to have started with the discovery in 1961 that Nb_3Sn can carry supercurrents of 10^5amp/cm^2 at fields of 100,000 oersteds and above. The difficulty with NB_3Sn is that it is very brittle and cannot be wound into coils in the ordinary way an electromagnet is constructed. In the past it has been produced in the form of very thin layers on ribbons of niobium or stainless steel, and then these were bundled into windings for electromagnets. Recently work has been proceeding at Brookhaven National Laboratory on a process for inserting rods of Nb_3Sn into larger copper rods, then drawing the rods down into wires and bundling them into cables. Verhoeven and Finnemore are working on a method for producing Nb_3Sn directly in the copper wire.

Imbedding the Nb_3Sn in copper has distinct advantages due to the strange properties of superconduction. The superconducting state involves coordination of the motions of conduction electrons in the material. The microscopic theory of superconductivity is based on the idea that a conduction electron attracts the positive ions that constitute the lattice of the material as it passes through them. This causes the positive ions in the lattice to move temporarily toward the track of the electron. The distortion of the lattice creates a region of relatively low potential energy for any other electron that happens to pass through the track of the first electron at the proper time. Thus, the response of the lattice to electron motion produces an effective interaction between electrons, coordinating their motions. A current induced in a closed loop of a superconductor (for instance, by withdrawing a permanent magnet from within the loop) will persist almost indefinitely, provided that the current is not too large. Persistent currents have been observed with no measurable decay for eighteen months. If the loop were not superconducting, the current would decay in a fraction of a second. This persistence is allowed only by the decreased energy of the lattice vibrations at the low temperatures of superconduction.

In a superconducting electromagnet, magnetic flux is forced to penetrate through the windings as the current increases. Because of imperfections in the material, these penetrations occur in abrupt jumps, called flux jumps. Flux jumps heat up the winding material, which may drive a portion of the winding resistive. If this happens, the current flowing through the normal resistive region will cause further heating, which may drive adjacent portions of the winding normal, thus causing an avalanche-type process. This may drive the whole magnet normal, breaking down the magnetic field and causing damage. To avoid these processes, the superconducting windings can be pressed into close contact with a low resistance metal, such as copper. If a portion of the superconductor is driven normal, the current flowing through it is then safely shunted through the lower-resistance copper, thus preventing an avalanche process. When the superconductor cools, the current is again diverted through it.

The goal is to maximize the area of the interface between the copper and the Nb_3Sn, not only to solve the avalanche problem, but also because the supercurrents concentrate within a few millionths of a centimeter from the superconductor's surface. Increasing the surface area of the superconductor thus increases the current-carrying capacity of the material.

The process Verhoeven and Finnemore are working on will use a directional solidification technique to produce a ductile wire of niobium dendrites imbedded in a copper matrix. A homogeneous alloy of Cu-Nb is formed into a wire approximately 70 mil (1.75 mm) diameter. The wire is passed through a tube which can contain the alloy in a molten state at temperatures of approximately 2000°C. The wire is then directionally solidified by a zone heating process in which localized heating causes a molten zone of about 1 cm long to pass down the length of the wire contained in the tube. If the zone moves at the proper speed and the convection currents are minimized, the niobium in the alloy forms into dendrites of a few microns in cross section. Figure 1 is a longitudinal section of one of the wires after directional solidification, showing the aligned Nb dendrites in the Cu matrix. Figure 2 is a cross section of the same sample, and each little cross is a primary dendrite. The number of dendrites in the 1.75 mm wire is approximately 50,000 and each dendrite is 2 to 3 microns wide. After the dendrites have been formed, the wire will be drawn down to a diameter of approximately 10 mil (0.25 mm) and coated with a layer of tin by drawing it through a molten tin bath. Heat treatment can then diffuse the tin into the alloy and form Nb_3Sn on the Nb dendrite filaments. This process will form a very large area of interface between the Nb_3Sn and the copper as is indicated by the cross section in Figure 2. The cables formed from such wires should be considerably more effective than the mechanically produced cables. When a continuous process of synthesis is developed the directional solidification method should be easier and much more economical. After more experience working with the materials, Verhoeven and Finnemore intend to develop a process using an alloy of niobium, copper and tin directly, thus avoiding the two steps of tin coating and heat treating to infuse the tin and form the Nb_3Sn.

Prior work with the directional solidification technique has shown that continuous rods of niobium two microns in diameter can be grown in an yttrium matrix as shown in the microphotograph in Figure 3. Similar results have been achieved in a thorium matrix and future development will involve alloying titanium with the niobium rods in the thorium or yttrium matrix. This may have some advantage over the current commercial method of mechanically inserting niobium-titanium alloy into a copper matrix for the production of magnet windings.

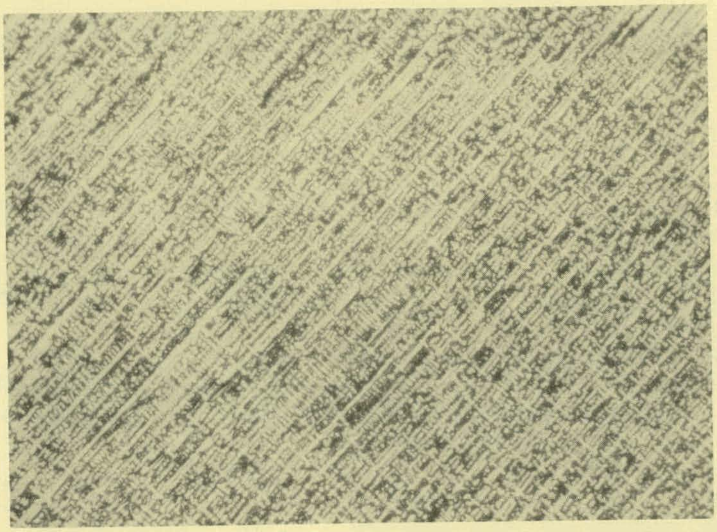


Figure 1. A longitudinal section showing aligned Nb dendrites in a Cu matrix. Magnified 238 times.

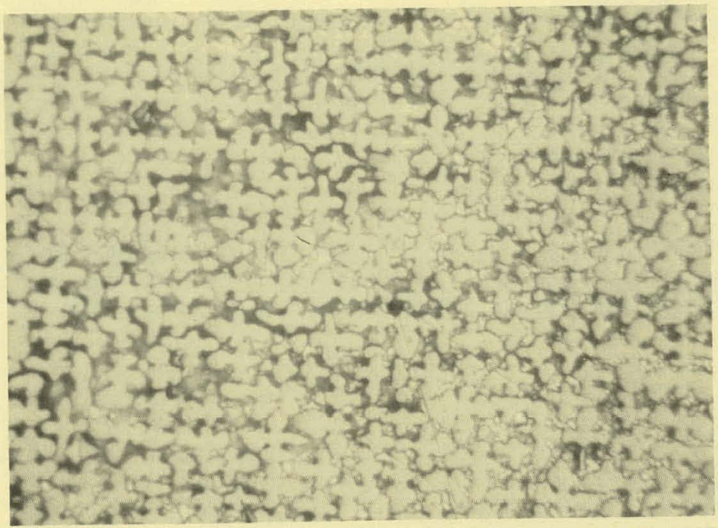


Figure 2. A cross section of the same sample as Figure 1. Magnified 792 times. Each small cross is a niobium dendrite between 2 and 3 microns wide.

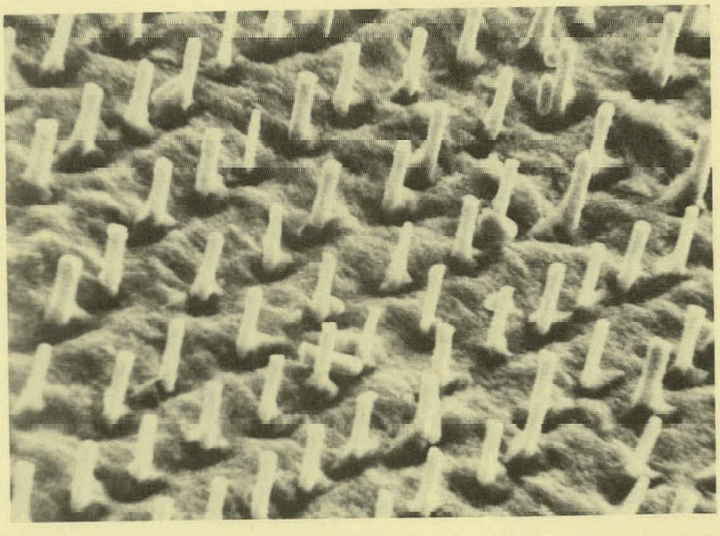


Figure 3. Niobium rods in a yttrium matrix. Each rod is approximately 2 microns in diameter. Magnified 1710 times.

Water Quality—A Greater Awareness

Water, composed of two of the most simple and abundant elements, hydrogen and oxygen, is the closest thing we know to a universal solvent. This characteristic makes it essential to life processes—the great majority of our own body weight is water. However, this same characteristic can also make water a conveyor of death when deadly poisons are dissolved in it. Water transports nutrients from the soil into plants and then dissolves and carries away part of the organic compounds thus formed when the plants later decay. Viewed from the perspective of water's solvency, the world's weather systems are one vast still, ever depositing the solutes of the earth in the oceans.

While some substances, such as common salt, dissolve quite readily in water, others, such as metals and many complex organic compounds, only dissolve slightly, or may only dissolve under special circumstances. In recent years some compounds and metals classically considered to be insoluble in water have been recognized as dangerous pollutants, some at concentrations of a few parts-per-million or only a few parts-per-billion. We actually know very little about the effects of trace organic pollutants, as it is only a few years since we have had techniques available for detecting them at such minute concentrations. In 1970, only ten organic compounds were known to be in drinking water samples taken from all over the world. By the end of 1975, more than 300 organic compounds were identified, and this number is increasing because of the rapid advance of technology for isolating and identifying them.

Researchers at Ames Laboratory became involved in establishing techniques for such trace determinations in the summer of 1970 when Dr. Vince Calder*, Dr. James Fritz, senior chemist, and Dr. Harry Svec, assistant program director, chemical physics, collaborated to solve the problem of an objectionable odor in Ames, Iowa municipal water. Using a "macroreticular resin" developed by the Rohm and Haas Company, Fritz and Calder managed to extract the offending material, then concentrated the sample. Svec and his associate, Gregor Junk, ran the sample through a makeshift combination gas chromatograph-mass spectrometer they had been working on to study interfacing problems.

From the first preliminary tests, it was clear that some of the compounds in the smelly Ames well were aromatic hydrocarbons. Eventually over 100 compounds were identified in the water from the well, many of which were characteristic of "coal-tar derivatives." These contaminants had been entering the Ames well via an aquifer that flowed beneath a waste disposal pit from an early twentieth century coal-gasification plant.

After using the new combination analytical technique to survey all twelve wells used to supply water to Ames, two of the most badly contaminated wells were taken out of service and the pumping schedules of the other wells were rearranged. There is no longer a taste and odor problem for the city's drinking water.

With support from the City of Ames, the ISU Water Resources Research Institute, the National Science Foundation, and the Department of Energy, these analytical techniques for identifying and quantifying trace organic pollutants were extended and refined. A flow-chart for the Ames Laboratory technique is shown in Figure 1.

A survey of thirty-five American and Canadian cities' drinking water was initiated using the sample collecting apparatus shown in Figure 2. The sample collectors were mailed to friends of the researchers along with detailed instructions on how to take the sample. The survey served to test the method and establish the magnitude of the problem of contaminated drinking water in large cities. The amount of organic pollutants in the drinking water of some of the cities of the survey was very low and indicated little or no potential problem. In a few cities, the amount of organic pollutants was much greater and the kinds of pollutants indicated at least a potential problem.

James S. Fritz

Harry J. Svec

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* Then a physical chemist with Ames Laboratory; currently with Rohm and Haas.

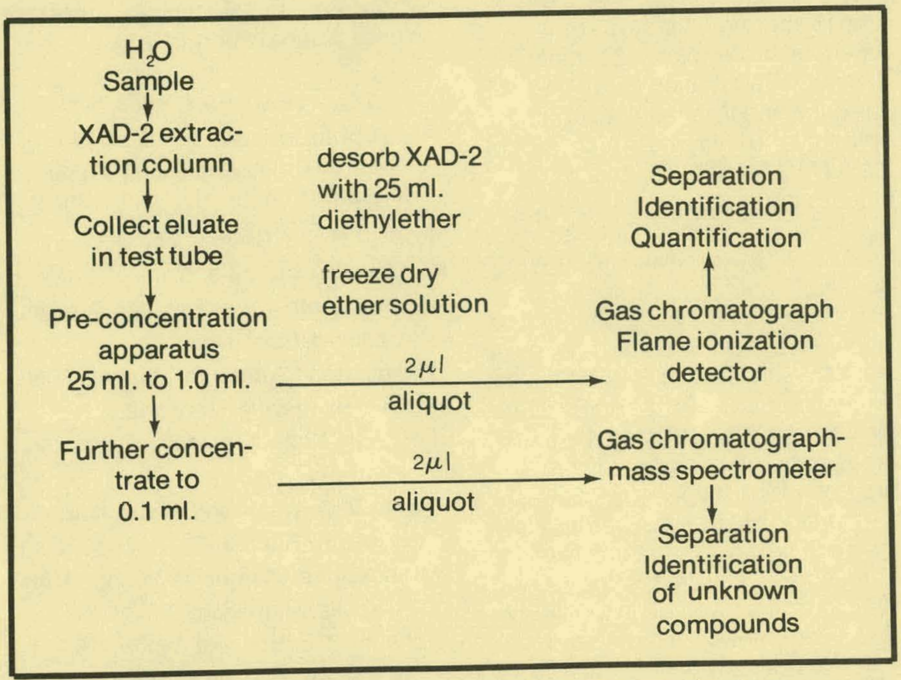
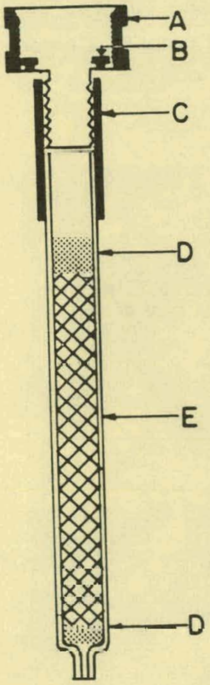


Figure 1. Flow-chart for extraction, separation and identification of unknown organic compounds in water.



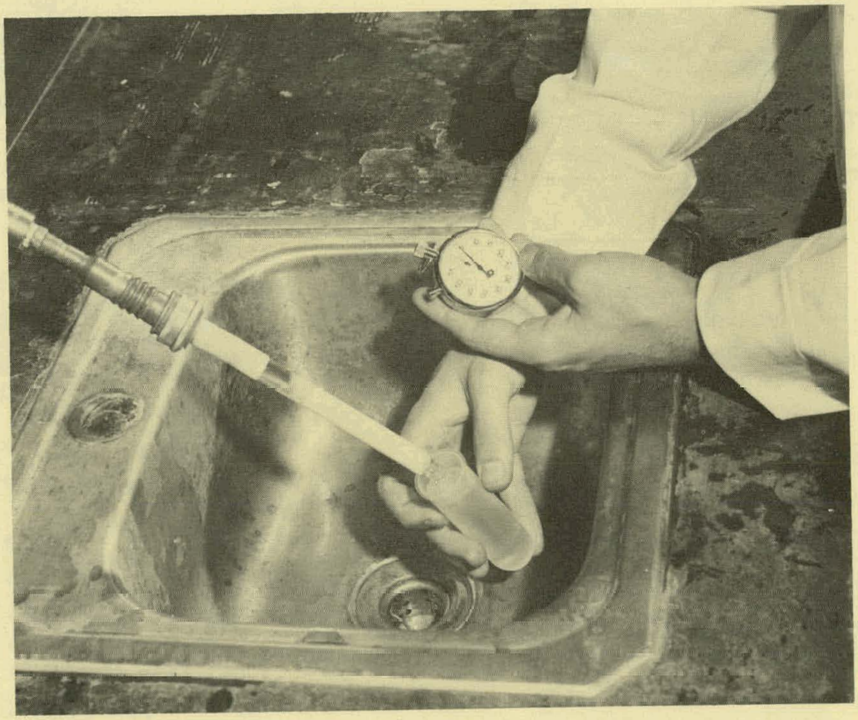


Figure 2. Scale drawing of apparatus for extracting organic solutes from finished drinking water samples. (A) standard garden hose coupling; (B) Teflon washer; (C) ½ inch I.D. Teflon tubing; (D) glass wool plugs; (E) ½ inch O.D. x 4 inch long glass tube packed with 5 ml, 40-60 mesh XAD-2 resin.
(Above) This sampling device with a standard garden hose coupling is used to collect field samples of finished water.

Application of these analytical procedures to a survey of water from rivers and wells at selected locations throughout Iowa from 1972 to 1975 revealed that all the major watersheds in the state are contaminated with varying amounts of agricultural chemicals. The compounds listed in Table 1 were all found in the waters. Their concentrations varied according to season and amount of rainfall.

The survey included three Iowa cities using deep wells as their raw water supply. Chemical contaminants in these waters were hardly detectable. However, water from shallow wells drilled in the flood plains of contaminated rivers contained the same chemicals that were in the rivers. Neither the filtering of water by seepage through the sand and gravel of the flood plain nor ordinary water treatment processes removes the chemicals. Even the use of activated charcoal or carbon, which some propose as a panacea for the problems of organic pollutants, is not always effective. In fact, saturated charcoal has been known to release sorbed pollutants back into the finished water.

Interest sparked by the results of this work led to the participation of Ames Laboratory in the current American Water Works Association Research Foundation study on occurrence and treatment of organic pollutants. The fourteen members of the AWWA listed in Table 2 are funding the study with assistance from EPA. Dr. Charles Oulman of Iowa State University's Engineering Research Institute is project coordinator for the three universities involved and Dr. Michael J. Taras is the AWWA Research Foundation's project manager. Fritz and Svec are the principal investigators for the Ames Laboratory portion of the project, which involves the determination of seasonal variations in the trace organic pollutants from the water supplies of the fourteen AWWA members involved in the study, and a comparison of the macroreticular resins with an activated charcoal method for removing the pollutants. The water samples are sent to Ames Laboratory in mailer kits like the one shown in Figure 3.

Table 2. Water Utilities Participating in American Water Works Association Study.

City of Santa Barbara, California
Metropolitan Water District of Southern California, Los Angeles, California
North Marion County Water District, Novato, California
Contra Costa County Water District, Concord, California
Washington Suburban Sanitary District, Hyattsville, Maryland
Louisville Water Company, Louisville, Kentucky
Miami-Dade Water and Sewer Authority, Miami, Florida
Minneapolis Water Works, Minneapolis, Minnesota
Pollution Control and Water Departments, Kansas City, Missouri
Owensboro Municipal Utilities, Owensboro, Kentucky
Manchester Water Works, Manchester, New Hampshire
City of Cleveland, Cleveland, Ohio
DeKalb County, Decatur, Georgia
Connecticut Water Company, Clinton, Connecticut

Table 1. Agricultural Chemicals Surveyed in Iowa Watersheds 1972-75.

Chemical	Type	Use
First two years		
atrazine	herbicide	weed control in corn fields
dieldrin	decomposition product of Aldrin (an insecticide)	
DDE	decomposition product of DDT	
Additional		
alachlor (Lasso)	insecticide	control root worm and some insects
propachlor (Ramrod)	insecticide	control rootworm and insects
2,4D	herbicide	control broadleaf weeds and brush
2,4,5T	herbicide	control broadleaf weeds and brush

Figure 3. Steve Sagerian, assistant research helper, fills and labels beakers with water study samples as Mike Arguello, post doc, displays a kit of metal tubes containing samples from a participating water works.

From experience with a bench-scale water treatment plant set up by the University of Illinois group headed by Dr. Vernon L. Snoeyink, it has been decided to establish a pilot plant using one activated carbon and a series combination of two resins. One resin was found to readily adsorb humic acids from water, the other removes chloroform; both resins can be regenerated. Under the supervision of Dr. John T. O'Connor of the University of Missouri, the Pollution Control and Water Departments of Kansas City, Missouri are installing the pilot plant to test these treatment methods.

No one knows the long range consequences of exposure to trace amounts of the combinations of chemicals being found in the drinking waters. There may be amounts below which there are no detectable adverse effects, or perhaps there is no safe level. Most of these compounds have not been closely studied for their health effects and certainly not in the combinations being found here.

The Ames Laboratory researchers have been able to enlist the aid of a colleague in the Department of Food Technology in preliminary tests for the possibility that some of these compounds might induce cancer. Dr. Bonita Glatz can perform an inexpensive test using *Salmonella typhimurium* bacteria to determine if given substances are mutagenic; that is, induce mutations in the bacteria. There is considerable evidence that, with few exceptions, chemicals which are carcinogens (cause cancers) are also mutagens (cause mutations). These tests, which were developed by Dr. Bruce Ames and his co-workers at the University of California at Berkeley, serve as an economical means of screening a large number of compounds for mutagenicity. Compounds that induce mutations can then be further pursued to investigate the possibility that they are also carcinogens. The test for mutagenicity can be performed for a few dollars, while an adequate test to determine if a compound causes cancers might cost several hundred thousand dollars.

The *Salmonella typhimurium* were developed with several mutations already present. The key defect is an inability to produce histidine, so that histidine must normally be added to their culture if the salmonella are to grow. This allows a very simple test to see if a chemical causes a mutation in the bacteria. If, in the presence of the chemical, the salmonella grow, the chemical has caused a mutation back to the ability to produce histidine. If the salmonella do not grow, the chemical is probably not mutagenic, as it has not induced a mutation in the bacteria. Figure 4 is a photograph showing both a negative and a positive response to the Ames test. The negative response shows a few colonies that have spontaneously reverted. In the positive test results, the sample (a disk that has been saturated with mutagen solution) is surrounded by colonies of bacteria that have been mutated.

Bonita A. Glatz

In addition to the histidine mutation, each tester strain of the salmonella contains two additional mutations that greatly increase its sensitivity to mutagens: one causes loss of the excision repair system and the other causes loss of the lipopolysaccharide barrier that coats the surface of the bacteria. The first assures that induced mutations will not be negated by repair back to the inability to produce histidine. The second allows large molecules that would be kept out by the coating to get into the DNA (genetic information carriers) of the cell.

Many of the organic compounds found in water in trace amounts by the Ames Laboratory studies are the consequences of human activity. Others occur naturally as water dissolves compounds formed by plant decay. These naturally produced contaminants can be transformed into toxic compounds as a consequence of treating the water for

negative positive

Figure 4. Negative and positive response to the Bruce Ames test.

human use. Chlorination of water that contains humic acids from soil runoff may create chloroform. Not only chloroform ($CHCl_3$), but also other trihalomethanes, bromodichloromethane ($CHCl_2Br$), dibromochloromethane ($CHClBr_2$) and even bromoform ($CHBr_3$) have been detected in chlorinated drinking water. The National Drinking Water Advisory Council is preparing to recommend that a minimum allowable concentration be established for these toxic compounds.

Ames Laboratory work has also shown that the concentration of various haloforms may change during the time between taking the sample and analyzing it in the laboratory. On one sample the chloroform concentration almost doubled in three days and then by the seventh day had decreased almost to its initial concentration. Similar but not identical fluctuations were found for the other trihalomethanes. For laboratory analysis, a system of stabilizing the sample with ascorbic acid was developed, but awareness of the dynamic nature of the trihalomethane-forming reactions is necessary in setting a pollution standard. Where should drinking water be sampled—at the treatment outlet, after standing in household pipes, or at some other point, in order to most accurately reflect the human consumption of these toxic compounds?

These analytical techniques are not applicable just to drinking waters. The solvency and ubiquity of water make these techniques useful in a wide range of applications. The fact that Ames Laboratory is one of the only two National Laboratories (along with Berkeley) located on a university campus has allowed researchers to participate in an interdisciplinary study to determine safe methods of pesticide disposal. The Energy and Mineral Resources Research Institute (EMRRI), ISU's arm that operates the Ames Laboratory, was one of seven departmental groups from Iowa State chosen by the EPA for the study. Along with the departments of Bacteriology, Entomology, Agronomy, Horticulture, Agricultural Engineering, and the Agricultural Experiment Station, EMRRI is performing the five year investigation for the Waste Management Division of the EPA. Chemical analysis and bio-assay checks are regularly run on samples taken from pesticide disposal pits at ISU's Agronomy farm and the Horticulture Station. Tests are conducted on persistent pesticides, such as chlorinated hydrocarbons, and nonpersistent pesticides—organophosphates and carbamates. Some of the persistent pesticides may last up to twenty years in the environment and are being gradually phased out. The nonpersistent pesticides, though more toxic, usually decay within a year. The main aim of the project is to find practical means for the safe disposal of the pesticides. Regular chemical monitoring allows tracking of the decay process and information on the bacteriological activity in the mixture of soil, rock and water contained in the disposal pits.

This entire series of studies has allowed a synergistic increase in the capability of the Analytical Mass Spectroscopy group of the laboratory. Back when Svec and Junk were investigating interfacing problems associated with gas chromatography and mass spectrometers, there were few GC-MS instruments available commercially. Companies marketing them did not release information on the interfacing system. There are now very good GC-MS systems available commercially that provide adequate information on the interface. Support from the National Science Foundation for the work on trace pollution in water allowed the purchase of a combined GC-MS. DOE Division of Basic Energy Science funds provided a data processing system to facilitate studies using that machine. More recently, application of GC-MS analysis to the study of stack gas from the City of Ames coal and solid waste fired power plant for the Division of Biomedical and Environmental Research of DOE provided funds for a new updated GC-MS system that will be connected to an improved data system supported by NSF through the water pollution studies. These systems will allow the rapid handling of large sampling requirements such as the extensive testing demanded by the AWWA study. The analytical task of such a large study would be prohibitive without these improved facilities.

Gregor Junk, assoc. chemist, and Loras F. Freiburger, Jr., Horticulture grad student, lifting a dirt and rock cover on a test chemical disposal pit at the ISU Horticulture station. Junk is coordinator on the EMRRI analytical chemistry team involved in the EPA financed, five-year, pesticide disposal study.

Application of these analytical techniques to new problems has produced some surprising results. A striking example comes from the analysis of stack gas from the Ames Municipal Power Plant (AMPP). Macroreticular resins, such as XAD-2, a polystyrene-divinylbenzene porous polymer, are effective not only in removing organics from water but also can sorb these compounds out of gaseous mixtures. It has been established that these resins can adsorb polynuclear aromatic compounds from air at concentrations as low as one part-per-billion. However, when these resins were used to remove the polynuclear aromatics (PNA) from the gases produced in the AMPP furnaces, very few or no PNA's were found. This result is unlike those reported by other workers examining extracts of incinerators and other combustion processes. The beginning of an explanation for this novel situation came only with subsequent attempts to extract PNA's from the fly-ash of the AMPP stack using solvents such as ether, benzene, tetrahydrofuran, n-pentane, etc., all of which were widely recommended in the literature. No PNA's were obtained although tests on soot from gas flames were successful. The clue to these difficulties lies in the nature of the fly-ash. The AMPP burns a mixture of 80 percent coal and 20 percent solid waste. The ash has a high silica content. It had been the practice to sluice the ash pit with water from the badly contaminated Ames well. Analysis of the sluicing water before and after contact with the fly-ash indicated an 80 to 90 percent reduction in organic contaminants (mostly aromatic and polynuclear aromatic compounds). Little or none of these aromatic compounds are recoverable from the ash by solvent extraction methods. It appears that the fly-ash has very strong sorptive properties for organic compounds. Investigations to determine the sorption mechanisms are proceeding. This is a most pertinent problem as additional power plants burning coal and trash combinations are anticipated in response to rising fuel costs.

Not all of the surprises have come from extensions of these techniques, however. An early surprise came during the initial study of the badly contaminated Ames well. Analysis of the extracts by infra-red spectrophotometry indicated a large number of compounds containing carbonyl $(-\overset{O}{\overset{\|}{C}}-)$, carboxylate $(-\overset{O}{\overset{\|}{C}}-O-)$, and ester $(-\overset{O}{\overset{\|}{C}}-O-R)$ groups. This finding was odd because these groups are not normally found with the compounds thought to be present in the smelly water. It was learned that these chemicals were present because of the manner in which the early sampling was done. For convenience, the resin sampler had been connected to a water faucet with a common nylon-reinforced plastic garden hose. Polyvinylchloride (PVC) hoses are made flexible by adding large amounts of "plasticizers." These are high boiling "esters" that are only slightly soluble in water. Yet, when the water from the well flowed through the hose, some of the "ester plasticizers" dissolved and were caught in the resin in the sampler. The plasticizers were responsible for the presence of the carbonyl, carboxylate and ester groups in the samples. These findings led to an expanded research project in which flexible plastic tubing manufactured for a variety of uses was studied to determine what organic compounds "leached" from the tubes when water or other fluids ran through them. The study identified a large variety of "esters" used as plasticizers, and other organic compounds that are added to the plastics to make them resistant to oxygen, ozone and ultraviolet light, to prevent the growth of molds, and to give them better surface characteristics. The additives found in nine different kinds of flexible plastic tubing are shown in Table 3.

Most of the compounds identified are relatively nontoxic, or no more so than the "fusel oils" present in highly colored or distinctly flavored alcoholic beverages. Some of them, however, have been shown to be highly toxic when ingested in large amounts.

Table 3. Total Amount of Organic Compounds Leached from Flexible Plastic Tubes.

Tubing Type	Concentration parts-per-billion
General Chemical Tygon	11
Laboratory Grade Tygon	4
Processed Milk Tygon	6
Food-Beverage Tygon	5000
FDA-USDA Approved Tygon	9
Hospital-Surgical Tygon	6
Green Plastic Garden Hose	170
Black Latex Rubber	26
Polyethylene	1
Polypropylene	2

It is not known if any of the compounds are toxic at levels found in the water. Awareness of these leaching problems has led a Minnesota plastics company to market a plastic tubing for beverage dispensing and medical uses made from a polyvinylchloride polymer with all the usual additives but with a thin inner lining of ethylene-vinyl acetate that is impervious to the passage of the additives into the fluids carried through the tube. A company representative documents the leaching problem with the Ames Laboratory publication that appeared in *Environmental Science and Technology,* **8** (1974) and indicates that the company cannot keep up with the demand for the tubing by medical facilities.

While the spectroscopy group has been extending its capability, the workers in Dr. Fritz's chemical separations group have been far from idle. Research is proceeding on the refinement of established techniques and the creation of new ones. John Ryan, a graduate student with the group, has been developing a system for using heat to desorb the pollutants from the macroreticular resins. This thermal desorbtion has some advantages over the use of ether to elute the sample from the resin. Evaporation of the ether during the solution concentration step may boil off some of the more volatile components of the sample. The ether also causes a

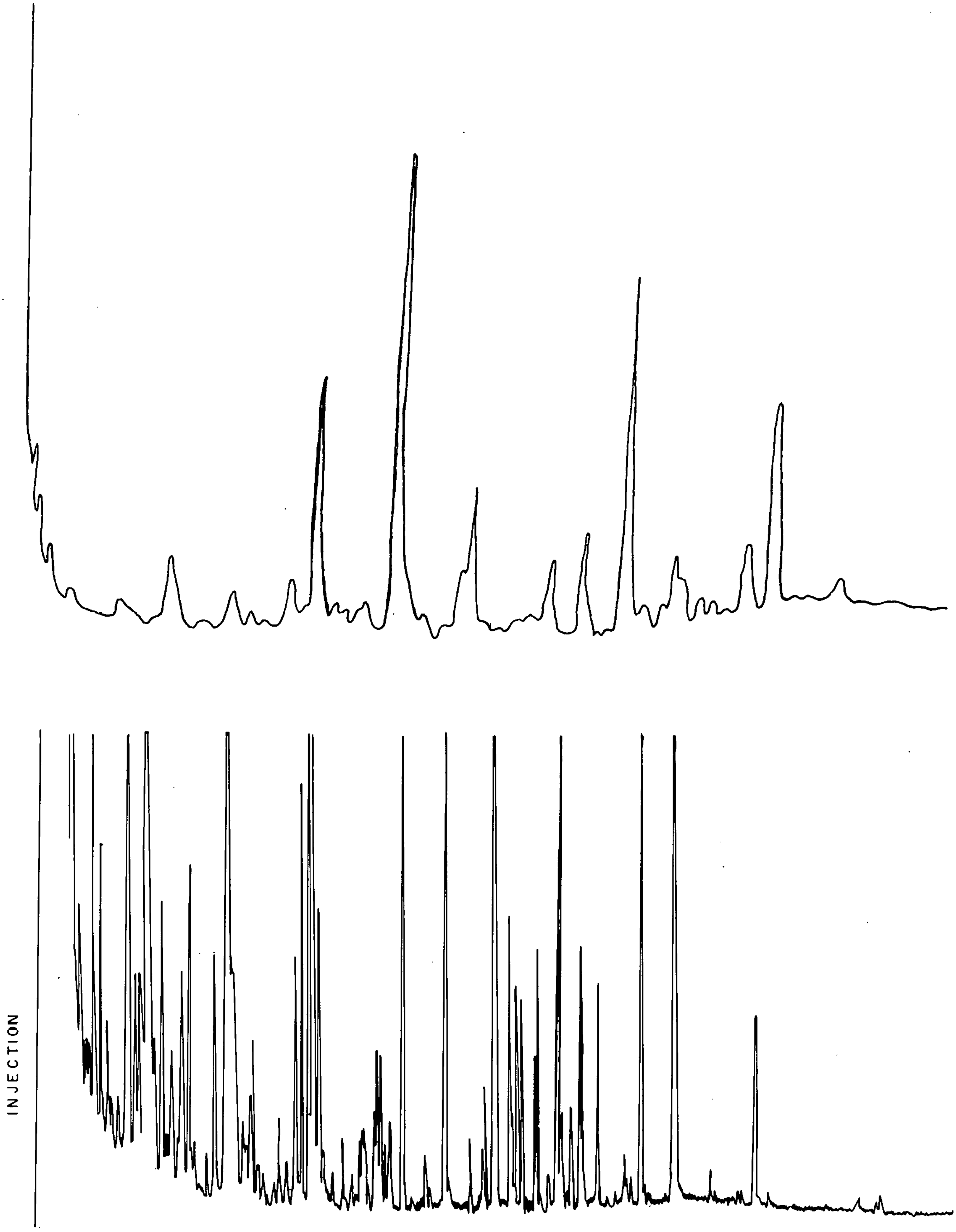


Figure 5. Packed column on top; capillary column on bottom. Organic material from Ames municipal water.

large peak that masks gas chromatograph detection of those low-boiling components of the sample that have survived the ether evaporation step. By using heat and helium gas to remove the sample from the XAD resin, and Tenax, a second resin, to assure that the sample enters the chromatograph as a "plug" and that no "noise" is caused by the introduction of water with the sample, the solvent problems are avoided and virtually all of the sorbed compounds are available for analysis. A considerably smaller volume of water can be passed through the XAD resin than in the original sampling procedure. This provides for simplified field sampling techniques using smaller resin chambers, an important consideration in the transportation of the samples from the source back to the laboratory for analysis. The size of the apparatus allows it to be sent through the mails quite economically. While this "miniaturized" version does not provide samples that are large enough for effective initial identification of a large number of unknowns, it is an excellent design for a regular, periodic monitoring program where the main variation is in quantities rather than in constituents of the sample.

Work is also proceeding on the use of capillary columns for the gas chromatographic separations. Ordinary gas chromatographic separation columns are of a large diameter and are packed with the stationary phase that performs the separation as the gas diffuses through it. Columns can be constructed of small enough diameter (hence the term "capillary") that packing is not required. The stationary phase coats the walls of the tube and separation occurs as the gas carrying the mixture of compounds passes through the column. Tremendous increases in resolution are possible this way, as can be seen from Figure 5, showing graphs of the same mixture separated by packed column and by capillary respectively. Packed columns can be made with up to 30,000 theoretical plates and capillary columns with 50,000 to 100,000 theoretical plates are now readily obtainable. Samples are resolved into 100, 200, and sometimes 300 or more chromatographic peaks, each peak representing an individual organic compound. In fact this method is so sensitive that the chromatograms are often too complicated for the average laboratory to identify more than a small fraction of the separated compounds, or to pick out and follow the concentrations of a few compounds of interest.

Such detailed methods are necessary, however, if the complicated mixtures of organic compounds from processes such as the production, refining, and combustion of fossil fuels are to be thoroughly described. The separations group is developing simplified procedures so that the tremendous power of capillary column gas chromatography can be used by an analytical laboratory of rather modest equipment and expertise. Gas chromatographic detectors that are selective for elements such as nitrogen, phosphorus, sulfur or the halogens are available but expensive. Two major approaches to the problem are being followed: first, the development of simple, quick liquid chromatographic fractionation methods so that each fraction will give a relatively uncomplicated chromatogram; and second, the creation of simple sorbent precolumns that will subtract all compounds containing a certain functional group from the final gas chromatogram. An example of the second system is a very short amine abstractor column that removes all amines from the sample.

TRISTAN—The Nuances of Nuclear Fission

Much remains to be learned before our understanding of the atomic nucleus is complete. Currently particles of small mass are theoretically treated as if the nucleons (protons and neutrons) move in orbits about the center of the nucleus, analogous to orbits of electrons in the atom. However, this model (called a shell model) does not adequately describe the behavior of heavier nuclei. Various so-called "collective" models are also used to characterize these heavier structures. These models are "collective" in that they describe coherent motions of the nucleus as a whole. As an example, regularities in the behavior of heavy nuclei in the region of the lanthanide series of elements suggest that the nucleus is deformed from a spherical shape and rotates about a minor axis. Nuclei also appear to be capable of a collective vibration, an in and out oscillation. While the shell model and various collective models are not necessarily mutually exclusive, their differences are an indication that we do not yet fully understand the interactions of nucleons in the nucleus.

While theoretical understanding of the nucleus is in need of further extension, there are also immediate questions of a practical nature created by our continuing use of the fission of ^{235}U or ^{239}Pu as a source of heat to generate electrical power. The design of power reactors that are safe, but are also highly efficient, requires an adequate map of the thermal energy distribution between the various products of the fission reaction. In addition, the design of control systems adequate to smooth out reactor power fluctuations requires a knowledge of the energy intensity and spectra of so-called "delayed" neutrons. A fission reaction releases neutrons that, when sufficiently slowed down by a moderator (such as ordinary water in a light-water reactor), produce further fissions. If additional fissioning were produced only by the fast neutrons that are emitted within a billionth of a second after fission, a stable reaction would not be possible because the rate of the reaction would fluctuate wildly. However, Nature is not often so unkind; not all the neutrons ultimately released are the immediate consequence of the initial fissions of the fuel. Some neutrons are released from fission products when these species further decay. On the scale of a billionth of a second, these lifetimes are long, varying from seconds to a minute. It is as if these neutrons were temporarily locked up or "delayed" in the unstable reaction products. Further fissions, produced by these delayed neutrons, contribute to a smoothing out of the rate at which the over-all reaction releases its energy.

As with most other specialities within physics, nuclear science develops through the interchange between experimentalists and theorists. Ames Laboratory experimentalists, Drs. John C. Hill and Fred K. Wohn, generate data on the behavior of various nuclear species and alert the theorists to areas of special interest. The theorists, Drs. James P. Vary and Stanley A. Williams, study these data in an attempt to formulate more generally

John C. Hill

Fred K. Wohn

James P. Vary

Stan Williams

applicable explanations of the physics of the nucleus, and in so doing suggest further characteristics of nuclei that can be investigated by the experimentalists. In this respect, nuclear scientists at Ames Laboratory are like all other nuclear scientists. They are unusual however, in that they are attacking both fundamental and practical problems by studying nuclear species so short-lived that special techniques developed at the Ames Laboratory are required to obtain the measurements.

In large part, understanding of the nucleus is derived from characterizing properties of nuclear excited states by measuring the radiations they emit. These excited states can be produced by means of both radioactive decay and charged-particle reactions. Radioactive nuclei near stability with long lifetimes (larger than a few minutes) allow the application of many techniques for the analysis of their properties. Much less is known about very neutron-rich and proton-rich nuclei far from stability, because these isotopes generally have very short lifetimes (a few seconds or less). While fairly easy to produce by thermal-neutron fission, such nuclei do not exist for long enough periods to be studied by the more usual techniques. They are in fact available for study only through such techniques as the on-line mass separator of the TRISTAN* facility at the Ames Laboratory.

The basic principles of on-line mass separation nuclear spectroscopy were developed in Scandinavia in the early 1950's when mass separators were connected to accelerators to study the products of charged particle reactions. Thus when TRISTAN began operation at the Ames Laboratory Research Reactor, under the direction of W. L. Talbert, Jr.,** in October 1966, it was the world's first isotope separator connected on-line to a reactor. The Scandinavian project resulted in the construction of a mass separator named ISOLDE*** at the European Organization for Nuclear Research, Geneva. The Ames project consequently was dubbed TRISTAN as a companion to it.

TRISTAN was designed to study the radioactive nuclei produced in the fission of uranium-235 after bombardment by neutrons emitted by the Ames Laboratory Research Reactor. A wide variety of neutron-rich nuclei are produced by the fission of ^{235}U as illustrated in Figure 1. This figure is a comprehensive view of the yield (production rate) of these fission products. The profiles shown on the left face of the figure illustrate the chemical nature of the products (as indicated by the proton number, Z). The back face profile relates the masses (indicated by the mass number) of the products. The profile is the sum of the slices shown separately for each fixed value for the proton number, Z. Each such slice, then, serves to indicate the yield of the various masses (various isotopes) of each different chemical element produced.

For each fission product element, one or more isotopes (different masses) will be stable while the remaining isotopes (parents) will undergo decay into daughters, which are isotopes of other elements. In general, the greater the difference between the mass of an isotope in question and the mass of the stable isotope (of that same element), the shorter the lifetime.

Krypton (Kr) and xenon (Xe) isotopes are easily separated from the remaining fission products, since they occur naturally as gases, and easily escape the ^{235}U sample. For these reasons, TRISTAN was first used to study these species and a long-range program was conducted to develop the techniques needed to study other fission product elements, including the non-gaseous reaction products.

The basic TRISTAN operation involves several steps. First there is need for "chemical" or element separation. This part is easy for the

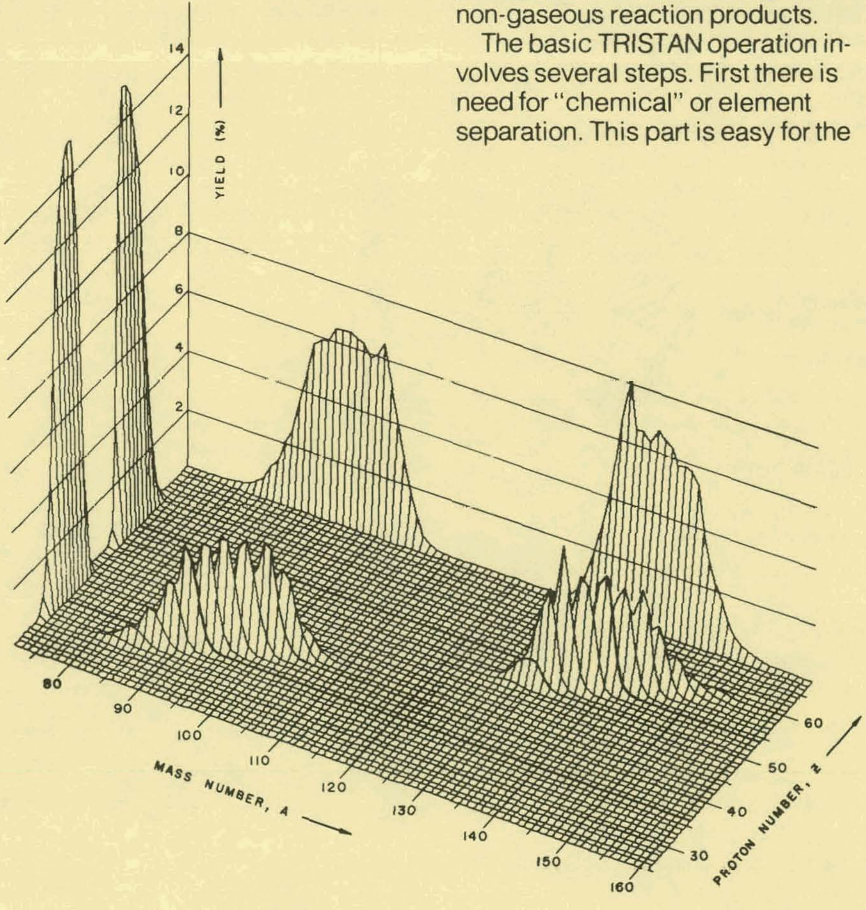


Figure 1. ^{235}U fission yield as a function of mass number and proton number.

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*An acronym obtained from **T**errific **R**eactor **I**rradiation **S**ystem **T**o **A**nalyze **N**uclei.

**Formerly Program Director, Nuclear Sciences; currently with Los Alamos Scientific Laboratory.

******Isotope** **S**eparator **O**n **L**ine, **D**anish **E**ngineering.

gaseous Kr and Xe, but considerably more difficult for the remaining fission products. The ^{235}U sample is in the form of uranyl stearate which is a yellow, soap-like powder. Neutrons emerge from the reactor via a hole or port (Figure 2) and irradiate this sample from which gaseous Kr and Xe rapidly diffuse.

Once this element separation has been completed, there is further need to separate each isotope of a given element from the other isotopes of the element. This is accomplished by the 90° sector magnet. The atoms of Kr and Xe are ionized in a hot plasma. As the ions pass through the field of the magnet, their paths are altered in proportion to their differing masses. Hence the magnet is referred to as the mass separator. Mass separation is necessary to select a particular isotope among the many isotopes of nearly the same mass. This is the heart of TRISTAN, and when it is connected directly to the mechanism which produces the radioactive nuclei, it is said to be "on-line" since there is no time-consuming intermediate step of collection and chemical separation.

The mass-separated atoms emerge from the separator in the form of an ion beam which can be directed into a collector placed near an array of detectors. The detailed study of a given isotope can then proceed without further time loss. This is crucial for studying those isotopes whose lifetimes are a few seconds or less. The entire process of fission, element separation, mass separation, and analysis of the products can proceed continuously. The short-lived nature of the nuclei does not deter the experiment, provided of course that the species survives the separation process and arrives at the detector before it decays. A photograph of the TRISTAN system is shown in Figure 3.

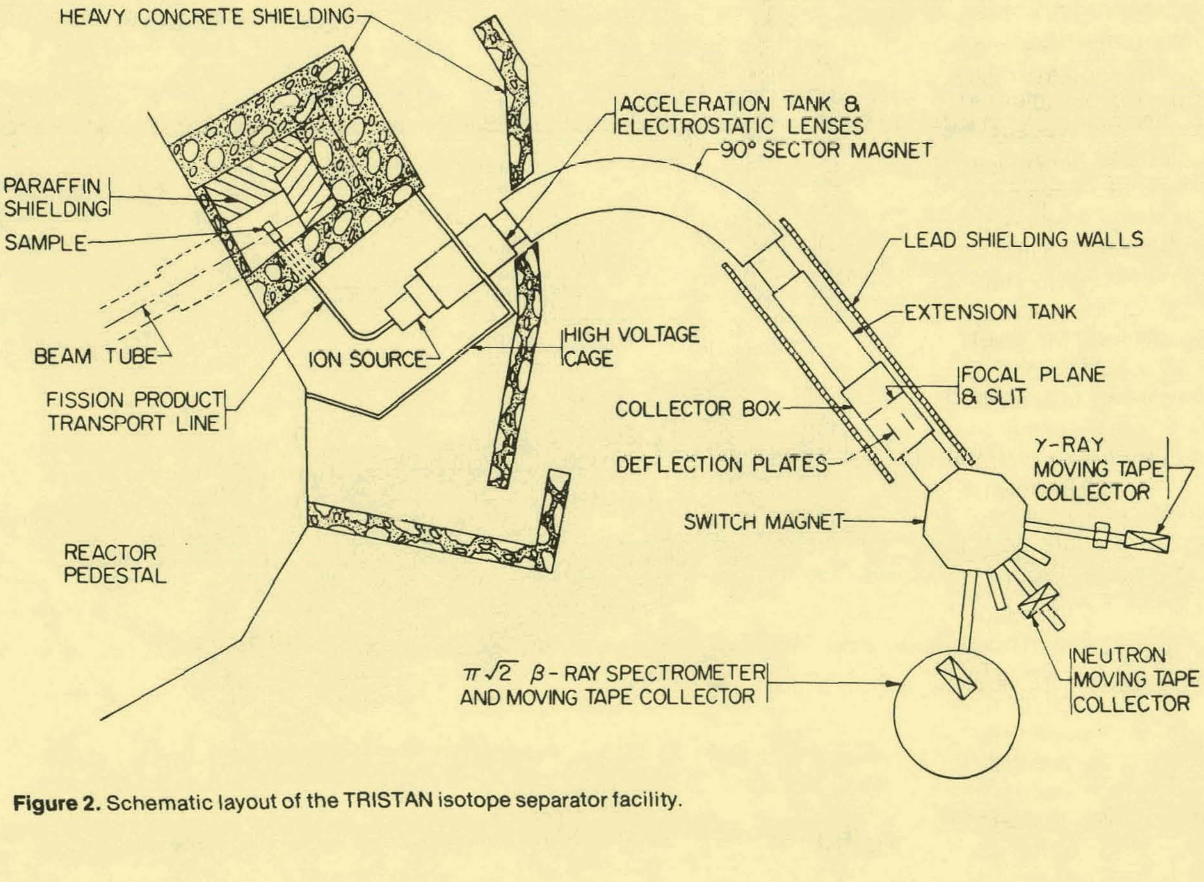


Figure 2. Schematic layout of the TRISTAN isotope separator facility.

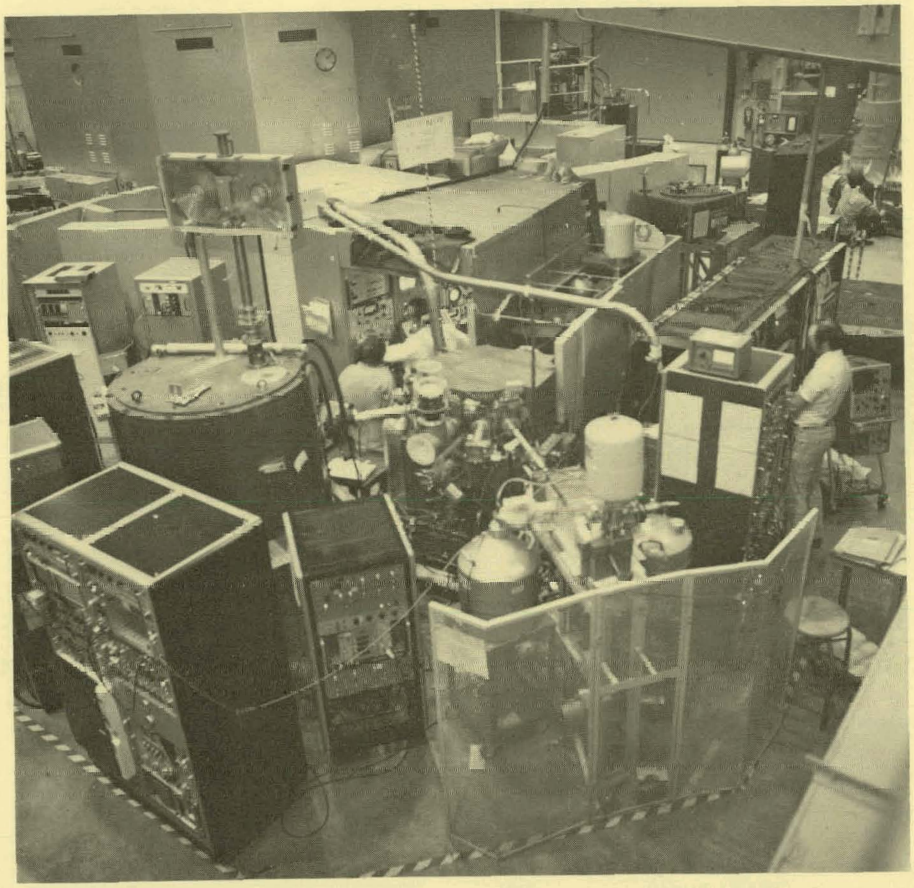


Figure 3. The TRISTAN system at work.

Among many innovations in the TRISTAN system, two of the more notable are the use of a switching magnet in the "on line" configuration to direct the ion beam to any of several detectors, and the use of moving tape collectors at the detectors. Activities of differing lifetimes are collected on the moving tapes. When the collection tape is moved with the correct speed, the short-lived activity recorded on one detector will have essentially all decayed away, leaving only the longer-lived activity to be counted by a second detector as shown in Figure 4.

Two major classes of studies are done at TRISTAN: survey and specific studies. The survey studies cover many nuclei; for each nucleus one measures the lifetime and the modes of its decay (emission of neutrons, electrons, and/or gamma rays). In particular the delayed neutron emission is characterized (this is important to power reactor stability and control) and the decay heat is measured (this information is important to safe reactor design, especially for calculation of reactor behavior following emergency shutdown).

The specific studies focus on specific nuclei formed in excited states. For each such nucleus quantitative measurements are made to establish what excited states it has, to determine by how much energy each is separated from the lowest energy (ground state), and to establish the angular momentum and parity of each excited state. The resulting energy level diagram provides a crucial test of a theory of internal motion and energetics of that particular nucleus.

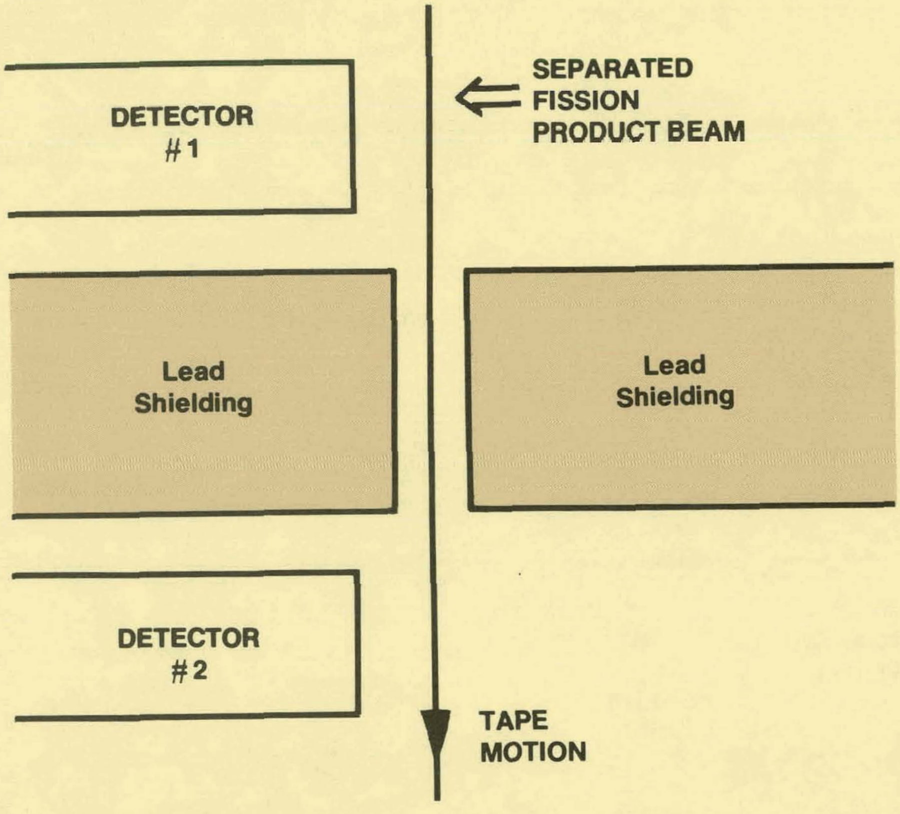


Figure 4. The use of moving tape collectors at the detectors.

The survey and specific studies that were conducted during the first ten years of TRISTAN are given in Table 1. These studies indicate the wide range of capabilities that have been gradually built into TRISTAN.

However, the scope of the original program was limited because only the gaseous fission products were made available in sufficient amounts for detailed studies. Only about 30 percent of the species produced by the fissioning of the sample could be analyzed; therefore a major effort was directed toward making other species available for study. Several techniques were tried over the years and the most successful one was selected.

With the introduction of this method, the utilization of an in-beam ion source, the system was rechristened TRISTAN II. The difference between TRISTAN I and TRISTAN II can be seen by comparing Figure 5 with Figure 2.

Table 1. Studies at TRISTAN I

Type of Study	Activities Studied
Survey studies	^{85m}Kr; ^{87}Kr; ^{88}Kr, Rb; ^{89}Kr, Rb, Sr; ^{90}Kr, Rb; ^{91}Kr, Rb, Sr; ^{92}Kr, Rb, Sr; ^{93}Kr, Rb; ^{94}Kr, Rb; ^{136}I; ^{137}Xe; ^{138}I, Xe, Cs; ^{139}Xe, Cs; ^{140}Xe, Cs; ^{141}Xe, Cs, Ba; ^{142}Xe, Cs, Ba; ^{143}Ba, La.
Specific studies	^{85m}Kr; ^{87}Kr; ^{88}Kr, Rb; ^{89}Kr, Rb; ^{90}Kr, Rb; ^{91}Kr, Rb, Sr; ^{92}Kr, Rb, Sr, Y; ^{93}Kr, Rb, Sr, Y; ^{94}Kr, Rb; ^{136}I; ^{137}I, Xe; ^{138}I, Xe, Cs; ^{139}Xe, Cs; ^{140}Xe, Cs; ^{141}Xe, Cs, Ba, La; ^{142}Xe, Cs, Ba, La; ^{143}Xe, Cs, Ba, La; ^{144}Xe, Cs.

Everything from the 90° sector magnet beyond the switching magnet is as before. The important difference is inside the heavy concrete shielding. Recall that in TRISTAN I a solid target of uranyl stearate was used, and the fission products were forced to find their own way out of the target into the transport line. There they entered

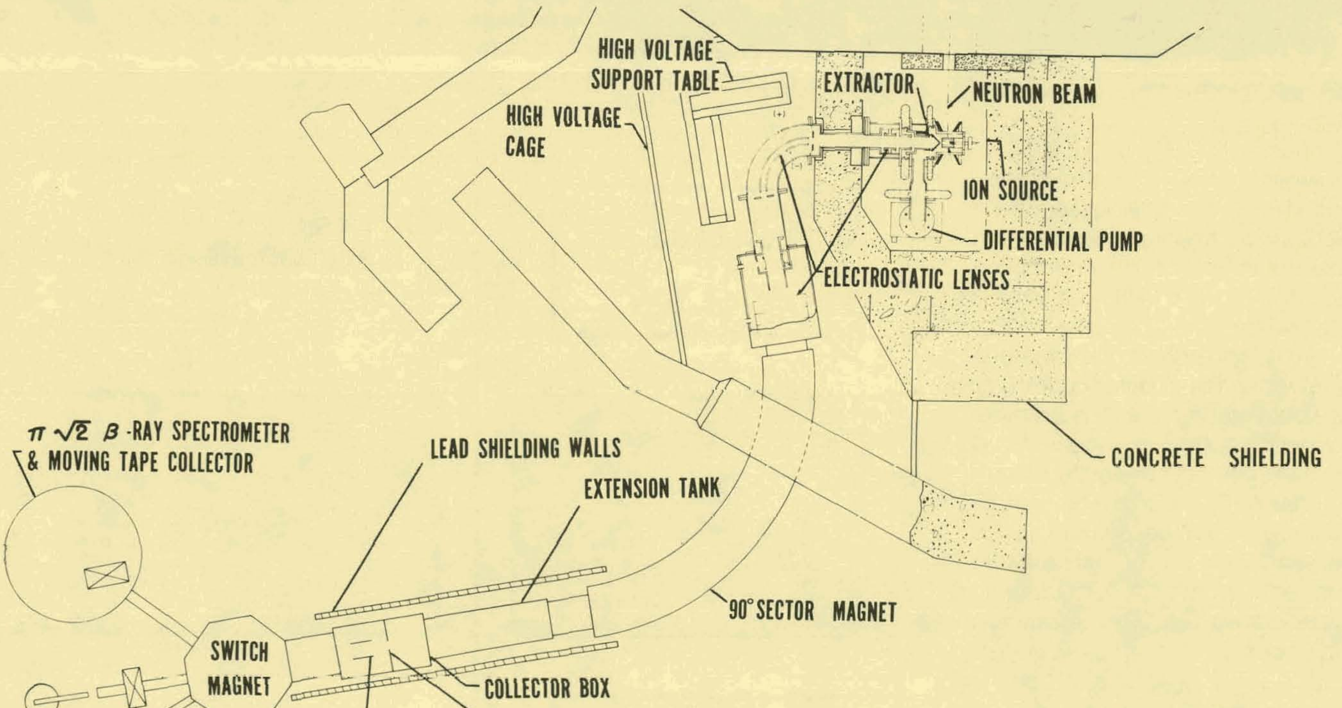


Figure 5. Schematic diagram of TRISTAN II in which uranium embedded in a graphite cloth is the ion source.

the ion source where they were ionized (charged) and accelerated into the mass separator magnet. Only the gases Xe and Kr, from among all the fission products, were able to make the journey on their own in large enough amounts for successful analysis. The "trick" was to create the other, less mobile, fission products directly as ions and to accelerate them out of the target area and into the mass separator.

This was by no means a simple feat, but was accomplished by using uranium imbedded in a graphite cloth as the target ion source. This target is in a chamber which is maintained at a very high temperature (1500°C) so that when the neutrons strike the target and create the fission products, a greater number of different species are released in a gaseous form. These are immediately ionized and accelerated by an extractor into the electrical transport system which bends the ion beam into the mass-separator magnet. From there the ions proceed as before. This new production and ionization system is the key to TRISTAN II.

Table 2 shows what has been accomplished at TRISTAN II in the short time since it came into being in June 1976. In addition to old friends krypton and xenon, there are now twelve different elements available in sufficient quantities for these studies.

The data base needed for efficient design of power reactors is weakest for the non-gaseous fission products, because few facilities can measure their properties and there are many more non-gaseous than gaseous products. The new capabilities of TRISTAN II will allow improvements in the data base where it is weakest. The DOE Nuclear Data Committee is considering increasing the upper limit allowed for the temperatures of the fuel in power reactors. This would improve the Carnot efficiency of power generation, but such decisions are only allowed by more complete knowledge of the distribution of decay heat. This same knowledge is essential for the safe disposal of reactor wastes, as these heat-producing decays continue long after the wastes have been removed from the reactors.

In addition to the immediate practical utility of these studies, the expansion of measurements on unstable species into new areas of the periodic table has a direct impact on the fundamental questions of nuclear structure identified earlier. The fission products made available by the capabilities of TRISTAN II are in an ideal area of the periodic table to allow comparisons with the nuclear shell models and collective models. For example, the ability to study nuclei near the very neutron-rich ^{132}Sn species (especially stable for such a heavy isotope of tin) allows theorists, Vary and Williams, to test the nuclear shell model in an entirely new area of the

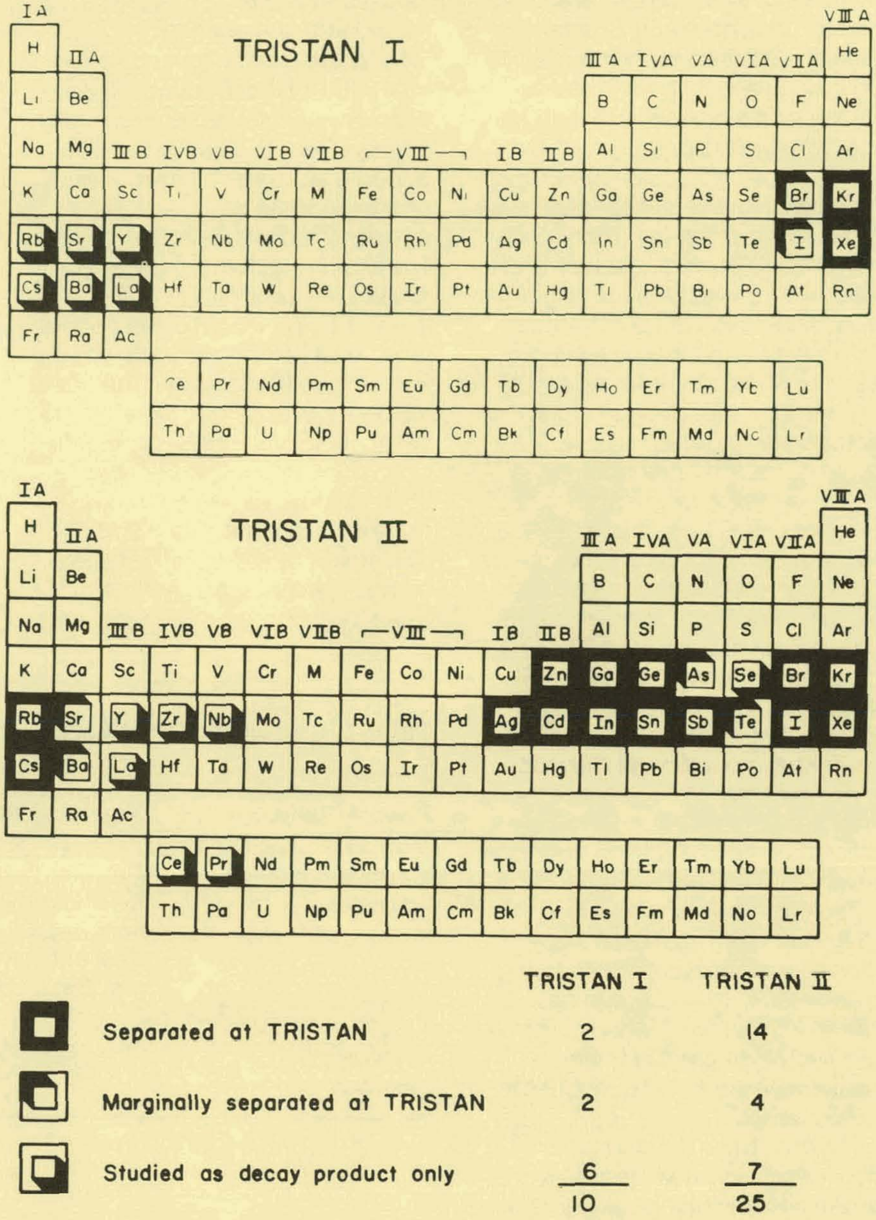
Table 2. Studies at TRISTAN II

Type of Study	New Activities Studied
Survey of emitted species	Zinc (Zn), Gallium (Ga), Germanium (Ge), Arsenic (As), Selenium (Se), Bromine (Br), Rubidium (Rb), Strontium (Sr), Silver (Ag), Cadmium (Cd), Indium (In), Tin (Sn), Antimony (Sb), Tellurium (Te), Iodine (I), Cesium (Cs), Barium (Ba)
First specific studies	^{78}Zn, ^{78}Ga, ^{118}Ag, ^{120}Ag, ^{122}Ag, ^{126}Cd, ^{126}In, ^{128}Cd, ^{128}In

periodic table. A quick reference to the periodic table (Figure 6) shows that the species available for study cover the region from silver (Ag) to lanthanum (La), the beginning of the rare earths. Isotopes of these elements will hopefully reveal crucial information for reconciling and unifying these two important, yet different, models of the nucleus.

While the theorists at Ames Laboratory are working in the traditional areas of nuclear theory in this exciting area of the periodic table, they are also working to bridge the gap between particle physics and traditional nuclear physics by the study of Relativistic Heavy Ions. The data for such studies come from experiments at the Bevalac facility at Lawrence Berkeley Laboratory, where experimentalists are slamming nuclei together at enormous energies. As the ability to perform such actions is quite new, it is not yet known exactly what to expect or what the consequences are. Some theorists suggest the possibility of "collapsed baryonic matter," a very dense complex with ultrahigh binding energies. Others propose the possibility of "pion condensates," a stable pion field existing at higher than normal densities. These studies address fundamental questions of the nature of strong interactions for the nuclear force and on a cosmological scale will provide insight into the nature of neutron stars and the processes of supernovas.

While the theorists give their attention to the minutia of the single nucleus as well as the vastness of stellar processes, a new chapter is about to begin for TRISTAN as experimentalists Hill and Wohn concern themselves with the more concrete details of moving the TRISTAN II facility from Ames to Brookhaven National Laboratory. When the Ames Laboratory Research Reactor ceased operation at the end of 1977, Hill and Wohn already were negotiating a move of TRISTAN II to Brookhaven to operate in what is termed a "users mode." The experiments will be conducted at Brookhaven and the data returned on magnetic tape to Ames Laboratory for analysis. They will establish a permanent users group



	TRISTAN I	TRISTAN II
Separated at TRISTAN	2	14
Marginally separated at TRISTAN	2	4
Studied as decay product only	6	7
	10	25

Figure 6. Periodic table of the elements, showing elements available with TRISTAN I and TRISTAN II.

Examining blueprints for the layout of TRISTAN II at the Brookhaven National Laboratory (BNL) are, from left, John C. Hill and Ron Gill, both of Ames Laboratory, Bob Chrien, BNL, Fred Wohn of Ames Laboratory, and BNL's Stan Ulc. (BNL photo by Rosen)

at Brookhaven so that other laboratories can perform experiments with the facility.

Two notable benefits (besides wider availability to the scientific community) offset the inconvenience of having the facility located away from Ames Laboratory. First, the neutron flux at Brookhaven is nearly twenty times as great as that of the smaller Ames Laboratory Research Reactor, thus the material produced for analysis will be correspondingly increased. Second, the facility will have two beam lines available which will allow two studies to be conducted simultaneously. The second beam line could be adapted to either delayed-neutron measurements or to dye-laser spectroscopy.

Even though about two-thirds of the elements produced in fission can be ionized and mass separated at TRISTAN, the ultimate goal is to separate all of the fission products. The rare earths and elements zirconium through palladium cannot now be separated because of their refractory nature. A major breakthrough in this field recently occurred with the demonstration at the University of Strasbourg, France, by Wohn, while on leave from the Ames Laboratory, and Dr. J. P. Zirnheld (Strasbourg) that a helium jet ion source can be used to separate all fission products. This ion source will be utilized on TRISTAN II at Brookhaven to greatly enhance the region of the periodic table available for study.

The new TRISTAN program at Brookhaven has been given a high priority by the U.S. Department of Energy and an in-house BNL staff of five new people will assume the responsibility for operating and managing the facility. This arrangement will free the Ames Laboratory Experimental Nuclear Sciences Group to devote their full attention to designing and carrying out new experiments at TRISTAN II in nuclear structure physics.

The Ames Laboratory Group will also be engaged in a new effort using the Bevalac facility at the Lawrence Berkeley Laboratory. This study will have as its goal the understanding of the nature of collisions between heavy nuclei at ultrahigh energies. The Bevalac is unique in that it is the only facility in the world that can accelerate heavy ions to energies as high as 70 GeV.

Recent renewed interest in alternative fuel cycles for power reactors increases the need for measurements of the properties of individual delayed-neutron emitters. Thus the techniques developed at the Ames Laboratory during more than a decade of operation should have a correspondingly greater impact in the future on both the practical and the fundamental questions in nuclear science.

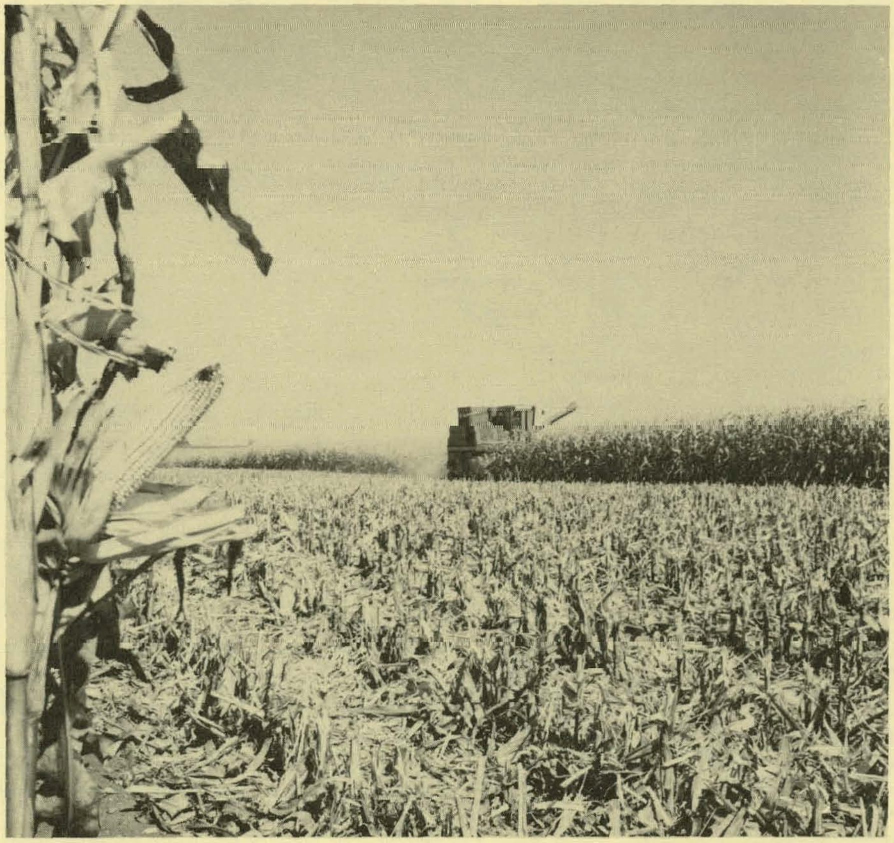
Revitalizing Iowa's Coal Industry

The Iowa Coal Research Project was established by the 1974 Iowa Legislature with a $3 million, 3-year appropriation to the Energy and Mineral Resources Research Institute (EMRRI) at Iowa State University. The Legislature hoped to provide Iowa with a means of meeting its energy needs and at the same time to revitalize the state's once-thriving coal industry.

At one time coal mining was a prominent industry in Iowa. However, as oil and natural gas became the dominant sources of energy in the U.S., Iowa coal production dropped from 9 million tons per year during the World War I boom when 18,000 miners were employed, to 600,000 tons of coal in 1976, produced by a workforce of less than 100 people.

Iowa currently imports about 96 percent of the energy that it consumes; about 80 percent of the imports are petroleum products and natural gas. As oil and natural gas supplies dwindle, Iowans are taking another look at the state's coal supplies.

The Iowa Geological Survey estimates that there are almost 20 billion tons of coal within Iowa's borders—enough to meet the state's energy needs for several hundred years. But, Iowa coal is a mixed blessing from an environmental standpoint. Although it has as high a Btu content as any other quality coal, its sulfur content is usually too high for it to meet state and federal environmental quality standards. Another special problem in the economic recovery and use of Iowa coal is that it underlies some of the world's richest agricultural lands. To tap this vast fuel reserve and at the same time preserve valuable farmland poses a unique recovery problem. During the past three years, the Iowa Coal Project has been working on solutions to these problems—not all have been solved, but many accomplishments have been realized.

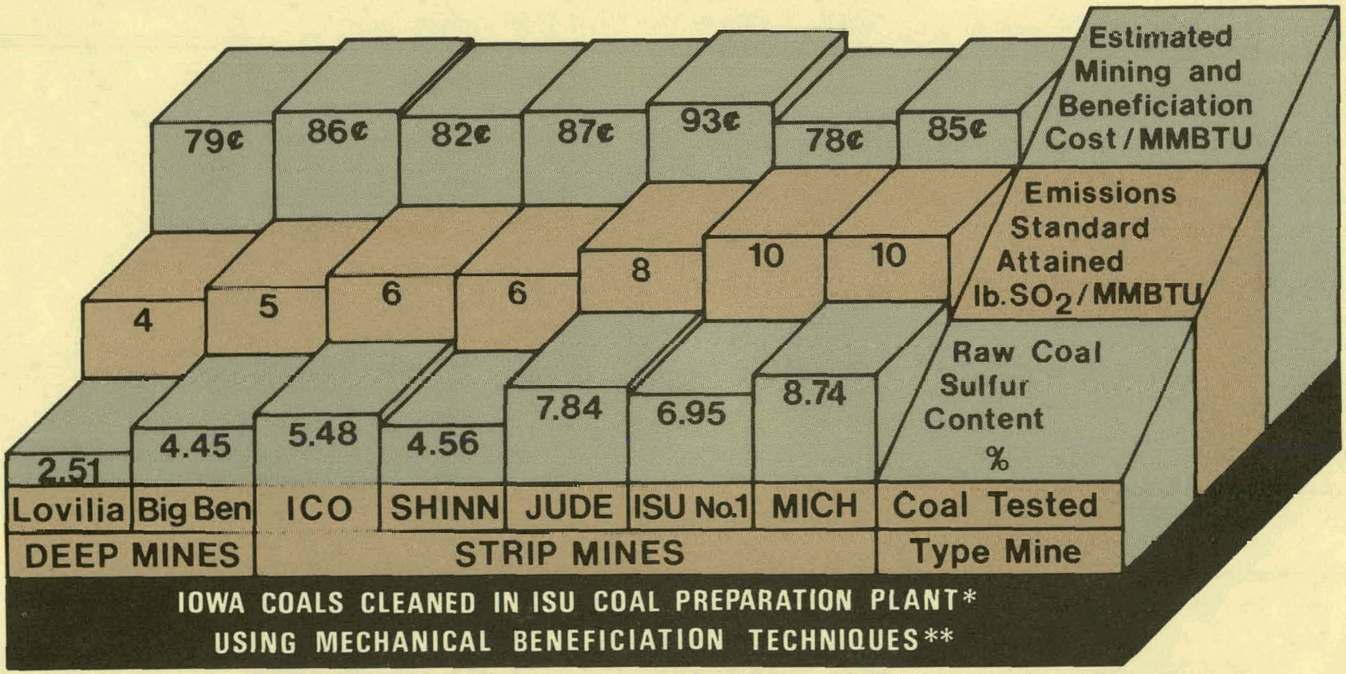


A coal-cleaning plant capable of processing 70 tons of raw coal per hour was designed and constructed on the Iowa State campus near the University power plant. Approximately 10,000 tons of coal from seven different Iowa mines were processed and evaluated for washability characteristics.

Iowa coal normally falls in the five to eight percent sulfur category. Generally, coals over seven percent sulfur will not meet an 8 lb SO_2/ million Btu input standard. Coal beneficiation results at the plant have shown that coals from Iowa's two existing deep mines can be physically beneficiated to meet emission standards of 5 lbs SO_2/ million Btu, and that coals from two of the five surface mines tested are capable of being physically beneficiated to meet a 6 lb SO_2/ million Btu standard. Eighty-five percent of the coal's heating value remains after processing.

Coal project personnel have determined that beneficiation will be required for the coal industry to develop within the state and be competitive with out-of-state producers.

A front-end loader moves cleaned coal from the stockpile at the end of the conveyor belt of the Iowa State coal-cleaning plant.



IOWA COALS CLEANED IN ISU COAL PREPARATION PLANT* USING MECHANICAL BENEFICIATION TECHNIQUES**

Type Mine	Coal Tested	Raw Coal Sulfur Content %	Emissions Standard Attained lb.SO_2/MMBTU	Estimated Mining and Beneficiation Cost / MMBTU
DEEP MINES	Lovilia	2.51	4	79¢
	Big Ben	4.45	5	86¢
STRIP MINES	ICO	5.48	6	82¢
	SHINN	4.56	6	87¢
	JUDE	7.84	8	93¢
	ISU No.1	6.95	10	78¢
	MICH	8.74	10	85¢

* Coals processed at specific gravity producing maximum sulfur reduction with highest possible yield.

** Heavy media and concentration table separations.

Results are for coal sized between 1½" and 45M.

Iowa Governor Robert Ray (center), Iowa State University President W. Robert Parks (left), and Plant Manager Dick Grieve discuss operations of the Iowa Coal Project's $750,000 experimental coal preparation facility.

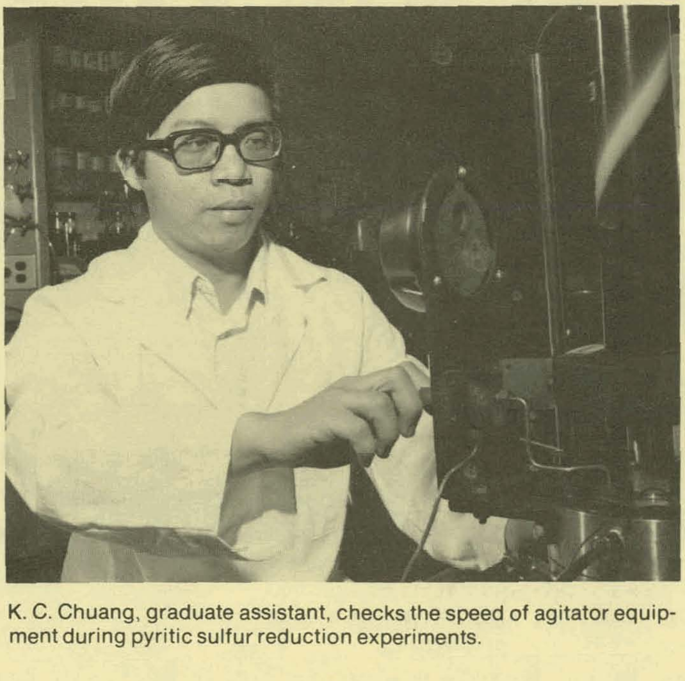
The cost of beneficiation would not be a deterrent to industrial development. Based on a per ton basis, the cost of beneficiation is less than the cost of importing coals to existing power plants that cannot be supplied by unit train.

This information has stimulated the establishment of a preparation plant industry in Iowa. Iowa utilities are also entering into contracts to purchase cleaned Iowa coal. However, further development of Iowa's coal industry will depend on improved transportation facilities, and continued research to reduce sulfur levels.

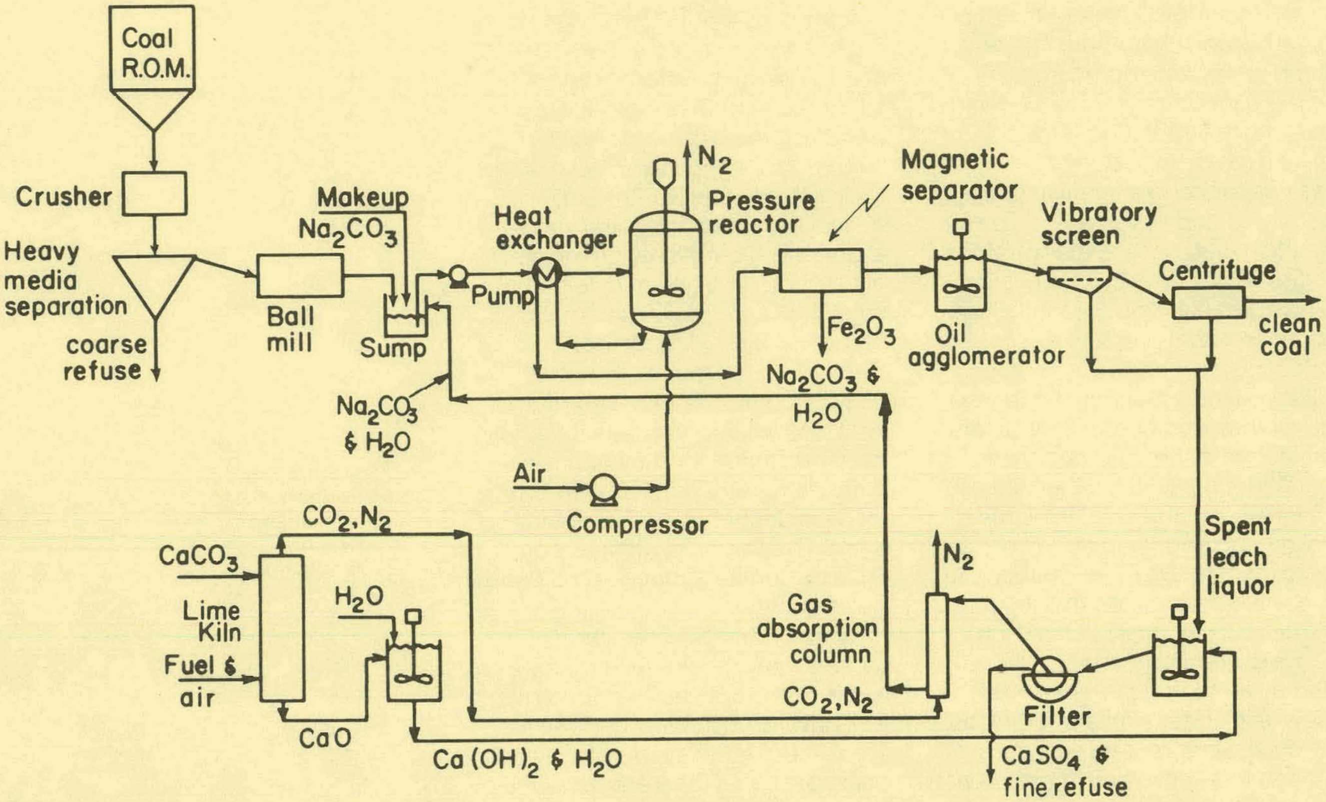
The Ames Laboratory, through its Fossil Energy Division established in 1977, is continuing its research to further reduce the sulfur levels in Iowa coals. Both mechanical and chemical methods are under investigation.

A number of methods for desulfurizing pulverized coal have already been tested: (1) treating coal with various oxidizing and reducing gases at elevated temperatures; (2) chemically extracting sulfur with various alkalis and acids; (3) chemically fracturing coal with liquid anhydrous ammonia; (4) physical cleaning of coal by selective oil agglomeration and froth flotation; and (5) various combinations of these.

The tests revealed that high-sulfur Iowa coals could be cleaned most effectively—as much as 95 percent of the inorganic sulfur was removed—when chemical desulfurization was combined with physical desulfurization methods such as gravity separation. Gravity separation removed a substantial part of the pyritic sulfur and other refuse, and chemical leaching removed most of the remaining inorganic sulfur, both pyritic and sulfate.



K. C. Chuang, graduate assistant, checks the speed of agitator equipment during pyritic sulfur reduction experiments.



Schematic flow chart of the proposed industrial process for desulfurization of coal.

Mining and reclamation go on side-by-side.

The Ames Laboratory beneficiation research has resulted in a proposed industrial process for desulfurization of coal as shown in the accompanying flow chart. Basically, after crushing and gravity separation, the fine-size coal is leached with a hot, dilute alkaline solution, containing oxygen under pressure. After cooling, iron oxides are removed by magnetic separation, and the coal is recovered by oil agglomeration, then de-watered. The alkali is recovered and recycled. The Department of Energy has provided funds for further development of the technique. The laboratory-scale project will be carried out in the State of Iowa's coal-cleaning plant where a portion of the feed stream can be diverted. In addition, various fine coal cleaning and recovery processes will be evaluated on a cost/efficiency basis.

Another Coal Project accomplishment was the integration of economic methods for mining and reclamation at the Iowa Coal Project Demonstration Mine #1 (ICPDM#1). Project reclamation goals included (1) characterization of Iowa overburden materials; (2) determination of overburden characteristics required to produce optimum post-mining agricultural production and evaluation of their long-term potential; and (3) development of methods for economically integrating mining and reclamation. To accomplish these goals, many field studies were conducted to evaluate soil erodability, slope stability, and procedures that promote root system development.

Data collected during the mining and reclamation operation indicate that reclamation costs would add approximately $2.50 to the total cost of a ton of coal mined at ICPDM#1; total mining and restoration cost is approximately $0.77 per million Btu. It was also determined that the mining methods for the ICPDM#1 project were competitive with alternative mining systems used elsewhere under similar conditions.

A computer program was developed to assist small mining companies with the development of good reclamation plans which comply with both Iowa and Federal reclamation laws. Computer models have also been developed to analyze the cost efficiencies of transportation and handling networks, and the competitive position of Iowa coal to coal from other regions of the Nation.

A multidisciplinary Environmental Team evaluated the environmental impact of surface mining on the ICPDM site to ensure that the mining was conducted in an environmentally acceptable manner. Preliminary results indicate that with proper planning before mining begins, most environmental problems can be prevented.

Agronomy studies were conducted on a restored three-acre site at ICPDM#1. The effect of various tillage techniques on production of row crops such as corn and soybeans, and response of these plants to different levels of nitrogen, phosphorus and potassium were examined. Corn plants grown on deep-tilled plots were up to two feet taller than corn grown on areas that had never been deep tilled.

Although the mining phase is completed at ICPDM#1, agricultural tests will continue until 1985 to develop sound, long-lasting agricultural practices for maximum crop production from reclaimed areas. Researchers will also continue to monitor surface and ground water quality.

Since preliminary research indicates that a shale-till interface at a four to one slope, as specified by Iowa law, is unstable when water saturated, further study will be conducted to see if different specifications are warranted.

A basic accomplishment of the Iowa Coal Project has been the development of a competent, interdisciplinary coal research staff and the facilities to make valuable contributions to the Nation's energy problems.

ICP reclamation worker, Doug Hertz, examines the 1976 soybean crop at the demonstration mine test site.

Midwest Reclamation Tour

The Midwest Reclamation Tour and Review group at the Iowa Coal Project demonstration mine site.

The purpose of a three-day Midwest Reclamation Tour and Review of mine sites in Iowa, Illinois and Missouri during August 1977 was to provide industry, state and federal agencies, and academic institutions the opportunity to review and evaluate reclamation technology at surface-mined coal lands. The group included representatives from the Environmental Protection Agency, the U.S. Bureau of Mines and the U.S. Department of Agriculture. State mine reclamation personnel from Iowa, Illinois and Missouri were also on hand. Other members of the group included representatives from Argonne National Laboratory and several universities.

The tour group visited the Iowa Coal Project (ICP) demonstration mine site near Oskaloosa on August 17. According to Dr. Stanley J. Henning, adjunct assistant professor of Agronomy at ISU and chairman of the tour, the techniques used at the ICP restoration project received many positive comments from the members of the group.

Post-restoration management was an important topic of discussion because federal law requires that mine operators in the midwest and east farm the site, or hire someone to farm it, for five years after the mining operation is closed down. The federal law requires mine operators in the west to farm the land for ten years after the mining operation is closed down.

A final report by the tour group includes topics covering research needs for restoration studies and how to comply with federal law as it applies to mining and restoration.

Nitinol—Remembers What You Want to Be

Dr. George Andreasen examines an orthodontic patient.

A revolutionary new orthodontic device that can make orthodontic corrections "from memory" has been developed by Dr. George Andreasen, Professor of Orthodontics at the University of Iowa. The wire orthodontic device is composed of a 55 percent nickel (Ni) and 45 percent titanium (Ti) alloy called 55-Nitinol and is the result of five years of laboratory and clinical research by Dr. Andreasen.

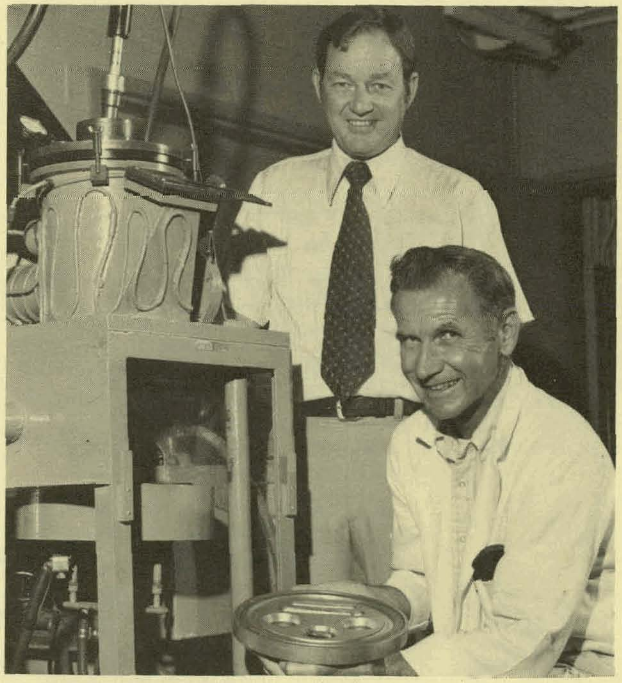
Because of its memory property and elasticity, the Nitinol orthodontic wire exerts a continuous force to move a tooth as far as required. By contrast, a stainless steel wire loses its elasticity or "goes slack" so that it moves a tooth only a portion of the prescribed distance. For this reason stainless steel wires have to be changed frequently as their force diminishes with displacement. Five successive stainless steel wires may be required to move a tooth during the course of treatment whereas a single Nitinol wire would have done the job.

Unitek Corporation of Monrovia, California, under license from the University of Iowa, has prepared the Nitinol orthodontic wire for market and field tested the product. Unitek's basic schemes for melting, fabrication and quality control of the wire were derived from the initial alloys and procedures developed at Ames Laboratory.

To establish the memory property for a specific shape, the Nitinol wire is first formed into a predetermined shape and then given a memory heat treatment between 800-1100° F depending upon alloy composition. After cooling the alloy to room temperature, it can be distorted into different shapes. With application of its transition temperature (about 90-110° F for an ideal dental alloy), the distorted wire will return to the shape that was established at the higher temperature. A different memory pattern can be established in the very same material by repeating the memory heat treatment for a different shape.

William Buehler of the Naval Ordnance Lab (NOL, now Naval Surface Weapons Center) first discovered the remarkable memory property in the 55-Nitinol material in 1962. As a result of correspondence with Buehler, Andreasen obtained a sample of one particular batch of the alloy, specifically Heat #9, from NOL. Andreasen experimented with the sample wire and found that it offered significant advantages over stainless steel wire currently used in orthodontics. However, when he had exhausted his supply, he found that he could not obtain additional wire from Heat #9. Other batches of Nitinol at NOL either did not have a memory activation temperature range that was suitable for orthodontic purposes, or did not have the desired elastic properties. Commercial firms were not geared to produce experimental quantities nor could they produce the material in consistent quality.

Unable to obtain the proper alloy commercially, in 1974, Andreasen contacted Frederick Schmidt and Charles Owen of the Materials Science Group at the Ames Laboratory for assistance with his materials problem. He confiscated a piece of the Nitinol wire from the mouth of one of his patients and submitted it to Ames Laboratory for analysis.



Frederick Schmidt (standing) and Charles Owen shown with the non-consumable arc furnace in which the Nitinol ingots were formed.

The analysis revealed that in addition to nickel and titanium, the carbon, oxygen and nitrogen content of the alloy were extremely important. Armed with this data on Heat #9, Ames Laboratory metallurgists prepared seven alloy ingots. Under the direction of Owen, the ingots were then fabricated into wire through a series of treatments whereby the wire was reduced to a final diameter of about 0.0205 inch (about 10 times the thickness of a human hair).

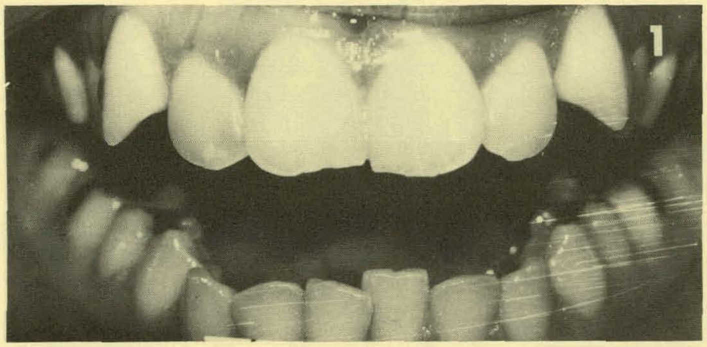
A piece of the Nitinol wire looks like a thin, lightweight plastic wire and to one's touch feels like ceramic material because of an oxide coating. Although the wire may appear to be delicate, it is extremely strong since memory activation causes heat energy to convert to mechanical energy.

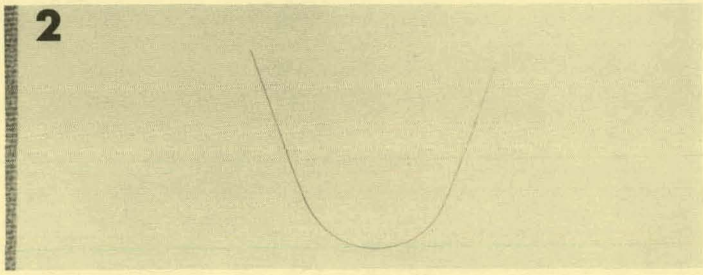
Force is the working tool of the orthodontist. Because of the consistent force applied when using Nitinol wire, fewer arch wire changes are needed for the complete treatment. This means that the patient's chair time and frequency of appointments are greatly reduced and less professional time is required.

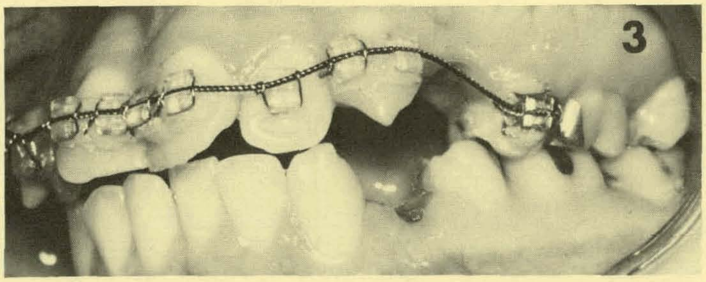
In general, there is little or no pain for the patient, only a feeling of pressure as the teeth are constantly and gently forced into their prescribed position. Because of the constant force and greater efficiency of the Nitinol wire, length of treatment time is reduced, in many cases to half the time of the earlier stainless steel technique.

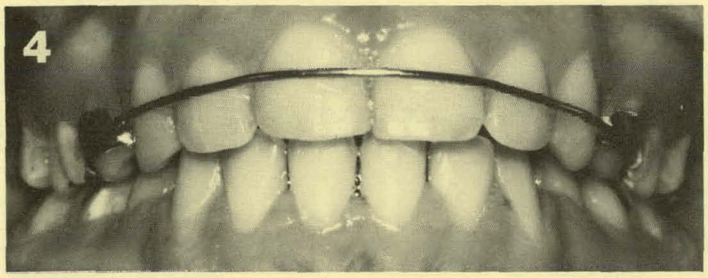
Both nickel and titanium are extremely noncorrosive and so resist attack by body fluids. The body, too, is fairly tolerant of the alloy's presence and does not tend to reject it.

The Nitinol orthodontic wire also offers many advantages to the orthodontist. Most important, the orthodontist will have mechanical predictability in his work. In addition, the wire device is convenient and easy to use. Since one or two Nitinol wires do the work of a wide assortment of different sizes of stainless steel wires, the orthodontist will not be required to have a large inventory of wires on hand.









1. Patient's teeth before brackets and wire insertion. 2. Nitinol heat-formed wire after setting "memory" at 800°C in a jig. 3. Patient's teeth with Nitinol wire at time of insertion. 4. Patient—end of treatment with retainer in place.

According to Andreasen, "Nitinol applications are limited only by time and imagination." He has dedicated himself to even further improvement of the orthodontic device on which he and the University of Iowa have filed for a use patent.

Since the quality control work, Ames Laboratory has also used the NiTi alloy in various applications. One of these has been the development of an expansion adapter which enables the resistance heating of wires and rods to high temperatures without distortion because of the metal's thermal expansion property.

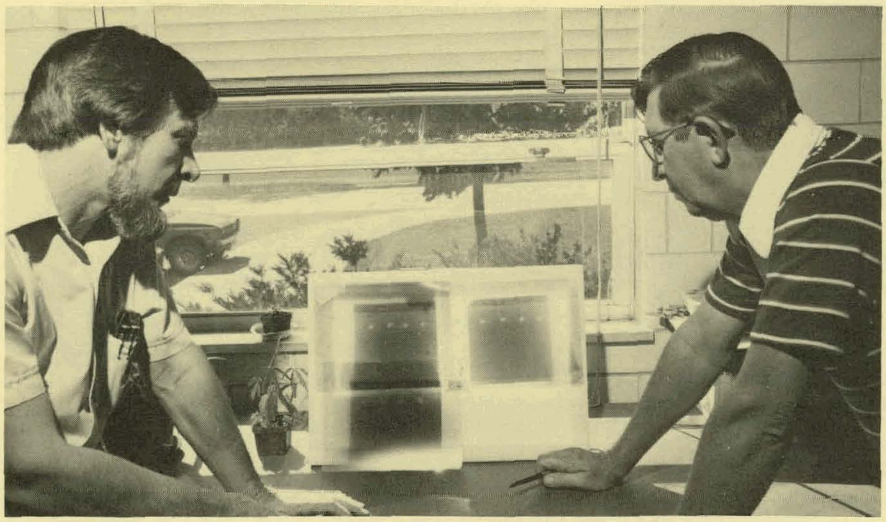
We can expect to see many other applications of Nitinol's memory and elastic properties in the future in medical and surgical devices, heat engines, weldless connectors, expandable structures, railroad hot boxes and any other type of device where mechanical efficiency is affected by heat.

Neutron Radiography—How Your Garden Grows

"How does your garden grow?" At least a partial answer to this fabled question may be provided as a result of Ames Laboratory studies.

Roland Struss, reactor mechanical engineer, Howard Taylor, agronomy, and Steve Willatt, formerly a visiting scientist from Australia, have been working to determine plant root growth rate, direction and concentration in corn and soybeans. Their studies involved neutron radiography techniques using two different sources of neutrons, one the Ames Laboratory Research Reactor (ALRR) and the other a californium source at the Sandia Laboratories in Albuquerque, N.M. Robert Jefferson, manager of Sandia's Nuclear Fuel Cycle Technical Development Department, assisted with equipment setup for the californium experiment.

In neutron radiography, a neutron beam is allowed to pass through an object and strike a metal transfer screen which becomes radioactive. More important, the transfer screen is "imprinted" with a radioactive image of the object. The image is made visible by placing the transfer screen in contact with a sheet of photographic film to expose it and then developing the film in the usual manner.



Roland Struss (right) and Steve Willatt study neutron radiographs showing seed development and root growth. The Ames Laboratory Research Reactor was used as a source for these neutron radiographs.

For the root growth studies, corn or soybeans are planted in rectangular aluminum cans. The higher concentration of water in the plant roots scatters the neutrons more than the soil. Thus, when the neutron beam passes through the aluminum cans in which corn and soybeans are sprouting, the roots show up lighter than the surrounding soil when the image appears on the negative.

Struss explained that with neutron radiography studies, the plants are not destroyed. The traditional methods for determining growth characteristics, he said, are nearly all of a destructive nature. The most common is that of washing the soil away from the roots—of course, this is a one-time test.

"Neutron radiography lets us continue measuring the same set of plants to determine growth characteristics. By using the same genetic material at different time intervals, we get better growth measurements," he said.

Also, because neutrons pass through the three-dimensional growing container, all the roots are seen. By contrast, roots grown under glass are distorted as they grow towards the glass walls of the container. Those against the glass are the only roots available.

Noting that neutron radiography is commonly used as a nondestructive test of industrial products, Struss said that application of the technique to the study of living things is a recent development. Industrial products, composed of solids such as metals and plastics are of much more varying hydrogenous density. As a consequence, neutrons are scattered in varying degrees and high contrast images are possible.

The main drawback in applying the technique to plant material is that water in the soil and the plant roots is the primary medium that scatters the neutrons. Because the plant roots themselves are mostly water and are small, they are only slightly more effective in scattering the neutrons than the surrounding soil, so there is very little contrast in the resulting image.

Photograph A shows the image contrast obtained when using the ALRR as a neutron source. Note that the roots on the 2-day old corn seed sprouts follow each other as if they were too "lazy" to find their own paths. Agronomists were aware of this phenomenon, but previously had been unable to watch it happen.

Pictures, as in Photograph B, taken during the californium neutron source experiment show no roots, but do show movement of the seeds. "In order to see the roots, we need either a bigger source of neutrons or better collimation," Struss said.

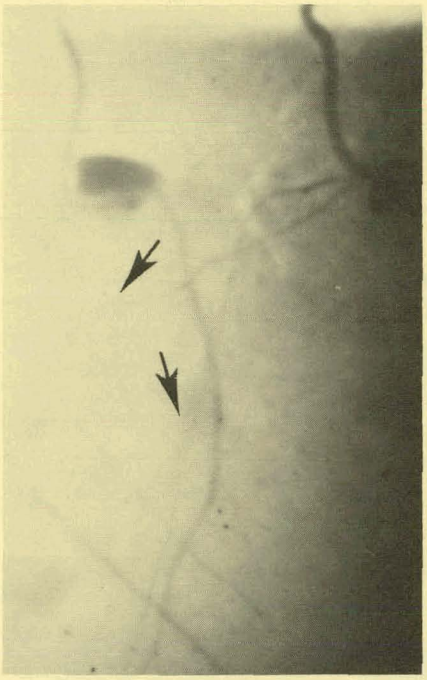
About six milligrams of californium were used in the experiment, but the test results indicated that 30-50 milligrams would be needed to produce acceptable neutron levels. This amount of californium would cost about $500,000 and would require 12 tons of shielding. Struss and Willatt had hoped that californium, a synthetic radioactive element, would be acceptable as a portable neutron source, but, obviously, it does not lend itself to field studies.

Collimation (reducing neutron scattering by making the beam as parallel as possible) is measured by an angle of divergence. The smaller the angle, the more effective the beam. Since the ALRR provided a beam with an angle of divergence of 6°, while the angle of divergence with Sandia Lab's californium source was 15°, the ALRR is more effective.

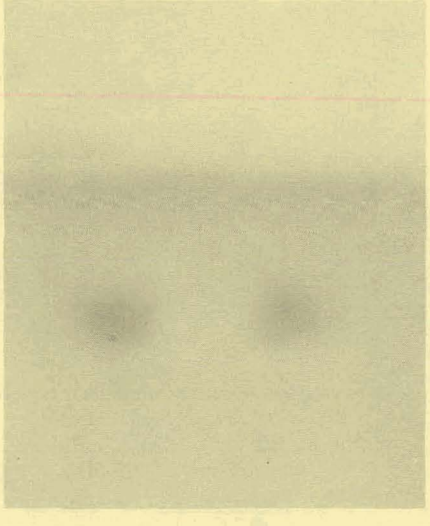
Struss and Willatt also varied the positions of the aluminum cans in the neutron beam, allowing a study of various exposure times. They found that the exposure time is limited because of plant growth which, after several hours, would blur the image.

The thrust of the work now is to further refine the neutron source technique and explore different films and photographic papers to gain higher contrast images. "I think we can show that this is an accurate laboratory technique for observing root growth," says Struss. "Right now there is no good way to get the necessary equipment into the field. The plants have to be grown in the aluminum cans so we can take them to a reactor," he concluded.

Although it is unlikely that scientists will ever use neutron radiography to tell you how your garden grows, the information gained from root growth studies will enable plant scientists to develop better plants that may very well wind up in your garden.



Photograph A. Image contrast obtained when using the Ames Laboratory Research Reactor as a neutron source.



Photograph B. Image contrast obtained when using the californium neutron source.

Fly Ash—A Future Ore Supply

Mike Murtha (left) and George Burnet discuss the Ames Laboratory high-temperature chlorination process for recovery of metals from coal fly ash. The furnace, as seen in the schematic, is open in the background.

With increasing energy demands, it is expected that the use of coal combustion for power will double by 1985, particularly as utilities comply with the 1975 Energy Policy and Conservation Act which requires oil and gas-fired plants to convert to coal. This means that sometime between 1985 to 1990, annual coal consumption could be about one billion tons; proportionately, about 100 million tons of coal fly ash would be produced. Fly ash disposal has plagued power plant personnel for years and could become an even greater problem in the future.

Ames Laboratory chemical engineers, Mike Murtha and Dr. George Burnet, are working on economical processes whereby the total fly ash material would have commercial value. Currently less than 15 percent of the fly ash material is being used primarily for construction and agricultural purposes. The remainder is buried or ponded as waste; both of these methods pose possible environmental hazards due to leaching of toxic elements.

The first step taken by Murtha and Burnet was to collect, from all over the midwest, samples of bituminous coal ash for characterization. Even though the fly ash composition of the various coals differed, the differences were smaller than anticipated. A representative coal sample was selected for the study.

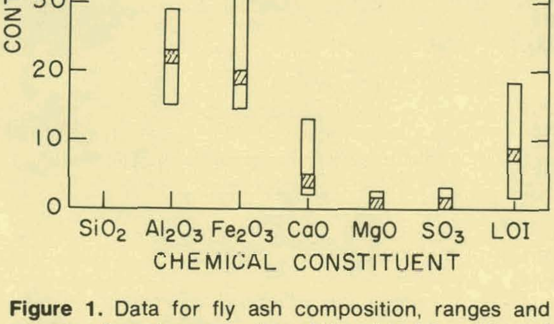


Figure 1. Data for fly ash composition, ranges and averages for a large number of fly ashes from different U.S. coals.

As shown in Figure 1, U.S. fly ash particles on the average contain approximately 22 percent aluminum oxide and 19 percent iron oxide by weight. Complete recovery of these metals would provide 90 percent of the U.S. aluminum requirements and 10 percent of the iron needs. The silica content of the fly ash can be used for Portland cement and glass. Fly ash aggregate would also be an asset to areas that have coal-fired power facilities, but shortages of natural aggregate materials.

At present, 90 percent of the aluminum ore for U.S. consumption is imported. Several large foreign ore fields have been nationalized and the price of aluminum ore has increased sharply in the past few years. Recovery of the aluminum in coal fly ash would be a conservation measure which would greatly reduce our dependency on ore imports.

Recovered iron could be used for the preparation of high-density media for coal and mineral beneficiation; seed material in high-intensity, high gradient magnetic processes for desulfurizing coals; and as iron ore. Demand for these materials and processes will increase as coal is increasingly employed and the need to mine and burn lower quality coal increases.

Murtha and Burnet have investigated two processes for recovering alumina (Al_2O_3) from fly ash—one a high temperature chlorination process; the other a lime-sinter process.

In the Ames Laboratory high-temperature chlorination process, the bulk of the iron oxide is first removed by magnetic methods, then a controlled amount of dry chlorine gas is passed through a bed of the nonmagnetic fly ash portion which is held at a high temperature (a minimum of 700° C). Solid carbon (C) is mixed with the ash in the bed so that the oxygen (O) from the metal oxides reacts with the carbon to form carbon monoxide (CO). Any iron from the oxide that slipped by the magnetic treatment and the aluminum react with the chlorine to form volatile chloride compounds. Because the chlorine reacts with the metal of greatest affinity first, the metals can be selectively removed; the iron is the most reactive and the silicon in the silica present is the least reactive. Figure 2 is a schematic of the bench scale experimental apparatus.

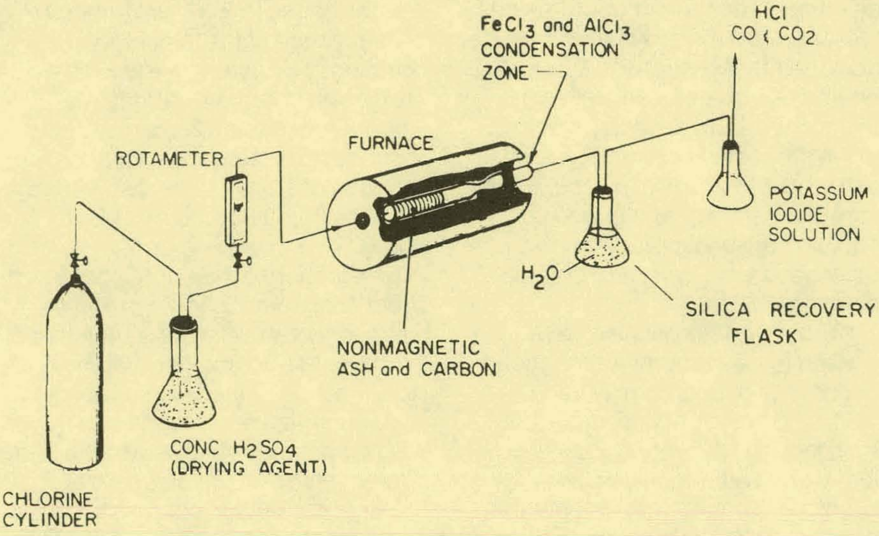


Figure 2. Schematic of the bench scale experimental apparatus used in the Ames Laboratory high temperature chlorination process for recovery of metals from fly ash.

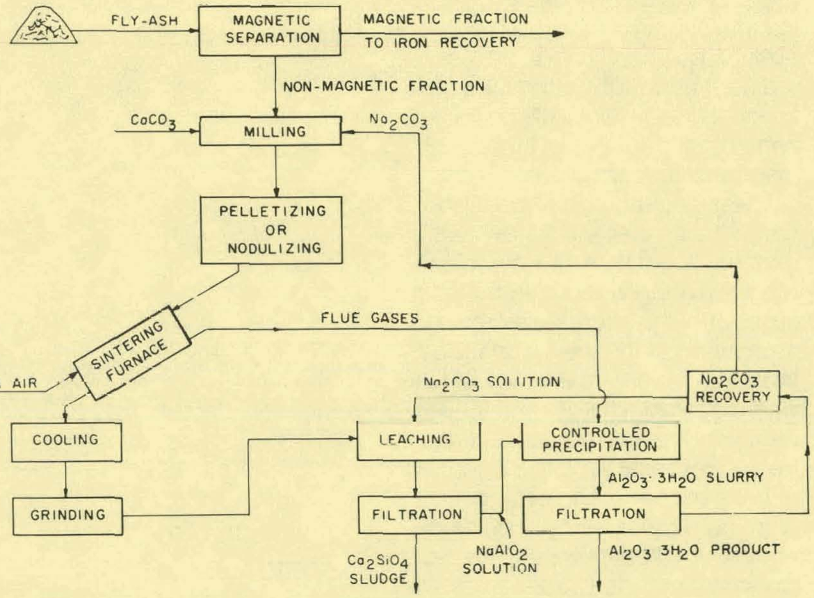


Figure 3. Extraction of alumina from fly ash by the lime-soda-sinter process.

Recovery of aluminum from aluminum oxide produced from bauxite ore requires a great deal of energy. Only 70 percent of this energy is needed to obtain aluminum from the aluminum chloride formed during the Ames Laboratory high temperature chlorination process.

Line-sinter alumina recovery processes are apparently successful in Poland and Hungary. Murtha and Burnet have improved the sinter process so that as much as 95 percent of the alumina can be recovered. They also have succeeded in cutting the energy required for the process by reducing the sinter furnace temperature from 1380° C to 1250° C.

In the Ames Laboratory lime-soda-sinter process, iron is first magnetically removed from fly ash. Next, limestone ($CaCO_3$) and soda ash (Na_2CO_3) are ground into the remaining non-magnetic portion, and the mixture is formed into pellets. After heating to 1200 to 1300° C in a furnace, the sinter mixture is cooled, ground, and leached with a sodium carbonate solution, as shown in Figure 3.

Previous results, both here and elsewhere in the U.S., have indicated that alumina recovery from the lime-sinter process is limited to 50 percent of that originally available in the fly ash. Insoluble calcium aluminum silicates are formed which reduce the amount of alumina available to be later precipitated as the final product.

By introducing sodium carbonate (Na_2CO_3) into the initial milling of non-magnetic fly ash with calcium carbonate, Murtha and Burnet found that alumina recovery could be increased to 95 percent. However, the calcium silicate sludge may contain too much sodium by this process for use in concrete, and will require additional treatment.

Lignite coals, like those found in Western coal fields, have higher calcium contents and would be better suited to recovery of alumina from their fly ash than the average bituminous coals because less limestone would have to be added to the process. For bituminous coal ash, about five tons of fly ash, eight tons of limestone, and one ton of sodium carbonate ground together before sintering will yield one ton of alumina.

Murtha and Burnet also have worked toward improved magnetic separation of iron from fly ash. Magnetic separation is important both for iron recovery and as the initial step in aluminum recovery. Harold D. Shank's instrumentation group at Ames Laboratory built a laboratory-size moving field electromagnetic device to be used in the magnetic separation studies.

The electromagnetic device will extract 70 to 80 percent of the iron from fly ash. Typical fly ash contains about 19 percent iron oxide (Fe_2O_3) by weight in the form of finely divided, dense, largely spheroidal particles of high magnetic susceptibility.

To take advantage of the high magnetic susceptibility, dry ash material is fed by a vibrator into the magnetic field where magnetically susceptible particles are drawn to and pulled along the magnetic surface. The magnetically susceptible particles agglomerate and fall into a receiver. The iron content of the extracted material is 55 to 58 percent and the contaminant levels of aluminum oxide and silicon oxide are about 10 percent each. In order to use the magnetic fraction as an iron ore, the combined contaminant levels of aluminum oxide and silicon oxide would have to be reduced to less than 5 percent. A microprobe is being used to find out if the silicon and aluminum oxides are actually separate from the iron particles. If the contaminants are separate from the iron particles, float-separation could easily reduce the contaminant levels.

When a process is developed which will reduce the silicon and aluminum oxides to levels of less than 5 percent, the next step will be to pelletize the product to provide a dust-free, transportable material ready for use in the blast furnace.

Utilization of the total high-iron fraction of fly ash as ore would reduce iron imports by about one-third. Ten million tons of iron could be recovered from an annual production of 100 million tons of coal fly ash as anticipated by 1985 to 1990.

In addition to the energy obtained from coal combustion, the metals recovery processes for alumina and iron would add another dimension to utilization of the very same coal and minimize ore imports and fly ash disposal problems. Murtha and Burnet envision metal recovery complexes built in conjunction with large coal-fired generating facilities as a common practice in the future.

The Whys of Grain Dust Explosions

Premo Chiotti

The increased frequency and severity of grain dust catastrophes in grain elevators prompted the Iowa Legislature to authorize an investigation, through EMRRI, into the causes of the explosions. A grain dust explosion group, formed in August 1975 under Dr. Premo Chiotti, senior metallurgist, found that three conditions must be present for grain dust explosions: a critical concentration of dust in a confined volume, oxygen, and an ignition source. Modern grain handling and storage methods increase the amount of dust in grain, and concrete elevators do not allow pressure build-ups to dissipate as did the early wood-plank elevators.

Chiotti and Mary Verkade, assistant chemist, compiled a literature survey and statistics on which to base research. Since instantaneous ignition sources have been reduced by good electrical practices and the introduction of inert gases into elevators to reduce atmospheric oxygen content had already been tested, the research group decided to concentrate their efforts on the "dust" component—the major thrust of their study would be to find a way to detect hot surfaces before they became hot enough to ignite grain dust (240° C for corn dust and 290° C for wheat dust).

Grain dust on hot surfaces emits particles ranging in size from invisible aerosols at low temperatures to visible smoke at combustion temperatures. A Pyr-A-Larm (R) smoke detector can detect both visible and invisible particles. To determine the minimum aerosol concentration level needed to trip the smoke detector, Edwin Gibson, associate metallurgist, and Virginia Morris, assistant chemist, used a condensation nuclei detector to measure the number of particles per cubic centimeter. Using a test apparatus which combined the smoke and aerosol concentration detectors, they simulated elevator heat conditions while varying the thickness of dust layers. Morris found that after the initial amount of dust required to produce aerosols, the concentration of aerosols escaping the heated surface drops with increasing dust layer thickness. This is because aerosols emitted at the hot surface stick to the dust particles on top of them. If the dust layer was of uniform temperature throughout, then aerosols would be emitted from the top dust particles, and thickness of the layer would not be a factor. Grain dust heated to pre-ignition temperatures also emits combustible gases. The gases can diffuse readily through any thickness of dust layer piled on a heated surface, possibly making them a more reliable indicator of hot surfaces. Gas chromatography tests were initiated to separate and analyze combustible gases given off by different grain dust sizes and types and to determine what part combustible gases play in the explosion mechanism. It is known that some combustible gases are emitted from heated grain dust at temperatures lower than those at which aerosols are involved; the majority of emissions, both gaseous and aerosol, begin in the 125-130° C range. A gas detector could be designed to react to a single gas emitted by hot grain dust, but which is otherwise not normally present in the atmosphere in significant amounts. Possible gases include H_2S, aldehydes, and ketones.

Virginia Morris draws a gas sample produced by heated grain dust. The apparatus is a specially designed tube furnace built in the Glass Shop.

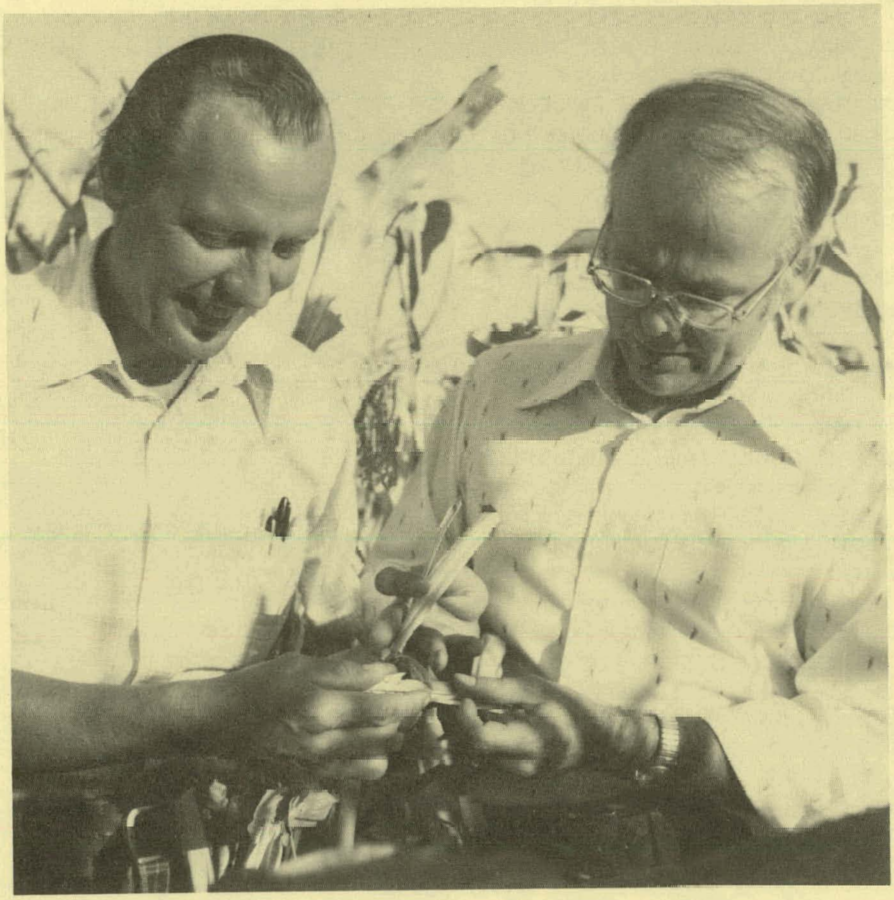
Cornstalks for Energy

A flatbed truck loaded with a stack of corn stalks drives by the coal stockpile on its way to the Ames Power Plant (in background). These corn stalks were burned at the power plant as part of a preliminary study on the feasibility of using agricultural residues as an alternative energy source. Agricultural residues represent a major unused national energy resource which has potential to form a compatible blend with coal for energy production. The Energy and Mineral Resources Research Institute (EMRRI) and the Engineering Research Institute (ERI) funded the study by Prof. Wesley Buchele, Agricultural Engineering, and Arthur Pohm, distinguished professor, Electrical Engineering, which covered the acquisition, storage and transportation of corn stalks, burning and determining heating value of the stalks, and studying the effects of stalk burning on stack emissions.

Initial tests at the Ames Power Plant revealed that dry corn stalks have a heating value of approximately 6500 Btu, which is comparable to that of the refuse currently being burned at the Ames Power Plant. The corn stalks shredded easily with no particular associated dust problem and readily moved through the plant system with complete combustion in the furnace. Blending the corn stalks with coal for combustion also dilutes the coal's sulfur emission.

As crop residue burning is envisioned, one-third of the total corn stalks produced in an area would be used for animal feed (more than enough to meet anticipated needs), another third would be used for fuel and the remaining third would be left in the fields for erosion control and soil enrichment. The researchers believe that as much as 60 percent of the coal burned at a power plant could be replaced by corn stalks and that the one-third portion of corn stalks in a highly developed agricultural area would be sufficient to fire a 500-megawatt facility. The corn stalks would provide a continuous renewable energy source; each farm community would be contributing to its own energy needs as well as reducing local and national energy import requirements.

That Fatal Attraction



Gregor Junk (left), Ames Laboratory associate chemist, and Jerome Klun, USDA entomologist, examine corn stalks in the field for evidence of corn borer activity. Klun and Junk are working toward the development of biological controls which could interrupt the insect's life cycle by confusing the sex communication system between adult male and female borer moths. Klun raised the moths and provided samples of the female sex pheromones and Junk and his colleagues used combination gas chromatography-mass spectrometry to assay the samples while using male moth response to emitted fractions as confirmation. They hope to refine the method and extend the study to natural habitat detection of smaller amounts of pheromones from a variety of insects. Crop damage caused by the European corn borer costs U.S. corn growers more than half a billion dollars annually.

Fingerprinting Coals

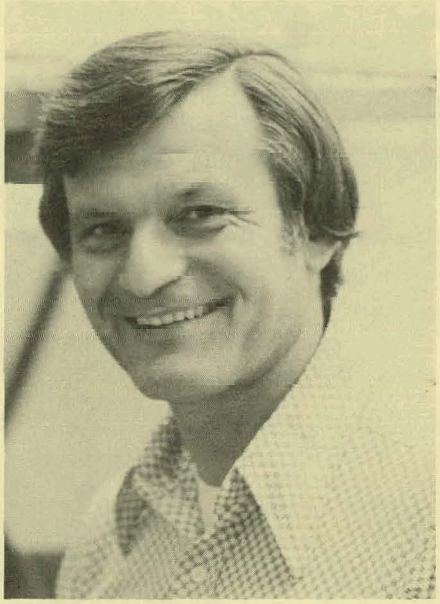
One important consideration in the types of chemistry which might be attempted in order to produce desired side products from raw coal with maximum efficiency, and, perhaps with minimum production of components such as polynuclear aromatic carcinogens, is the average aromatic ring size in raw coals. Two measurements which can help make this determination are the ratios of aliphatic to aromatic protons, and aliphatic to aromatic carbons in solid coals (a consideration of these ratios for the compounds benzene, naphthalene, and methylnaphthalene will help make this point clear).

One, and perhaps the only, method of fingerprinting aliphatic and aromatic carbon and hydrogen in solids is nuclear magnetic resonance (NMR). Both ^1H and ^{13}C exhibit absorption of radiofrequency excitation in high magnetic fields, and the frequency of the absorption can characterize the chemical environment of the nucleus in question.

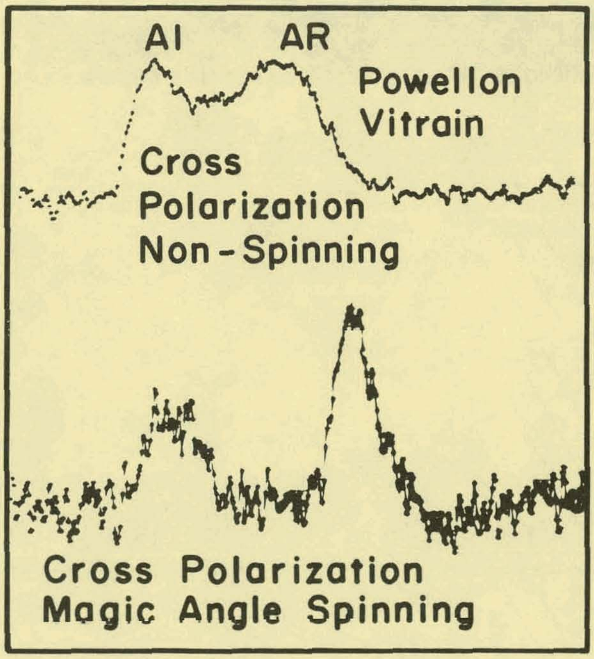
A problem complicating a determination of such "chemical shifts" of NMR frequencies is that coupling with neighboring nuclei broaden resonance lines to the extent that chemical shifts are unobservable. In the case of ^{13}C, coupling to ^1H dipolar fields is the major cause of the broadening. Even when this broadening is removed, however, residual broadening associated with the anisotropy of the chemical environment in solids can still mask chemical information.

Recent experiments by Dr. Bernard C. Gerstein, Ames Laboratory chemist, have used combined strong heteronuclear decoupling to remove the effect of the ^1H on the ^{13}C NMR line, and magic angle spinning to remove broadening due to anisotropic chemical environments in solids. Resultant ^{13}C NMR spectra of coals show a clear separation of the peaks associated with aromatic and aliphatic carbons in coals, as shown in the accompanying figure, in which strongly decoupled ^{13}C spectra without (top) and with (bottom) magic angle spinning are exhibited.

With this resolution, it will now be possible to follow processes such as hydrogenation of solid coals under relatively gentle treatment utilizing NMR. Additional resolution in similar experiments on ^1H in solid coals will allow a determination of average ring size in such species.



Bernard C. Gerstein

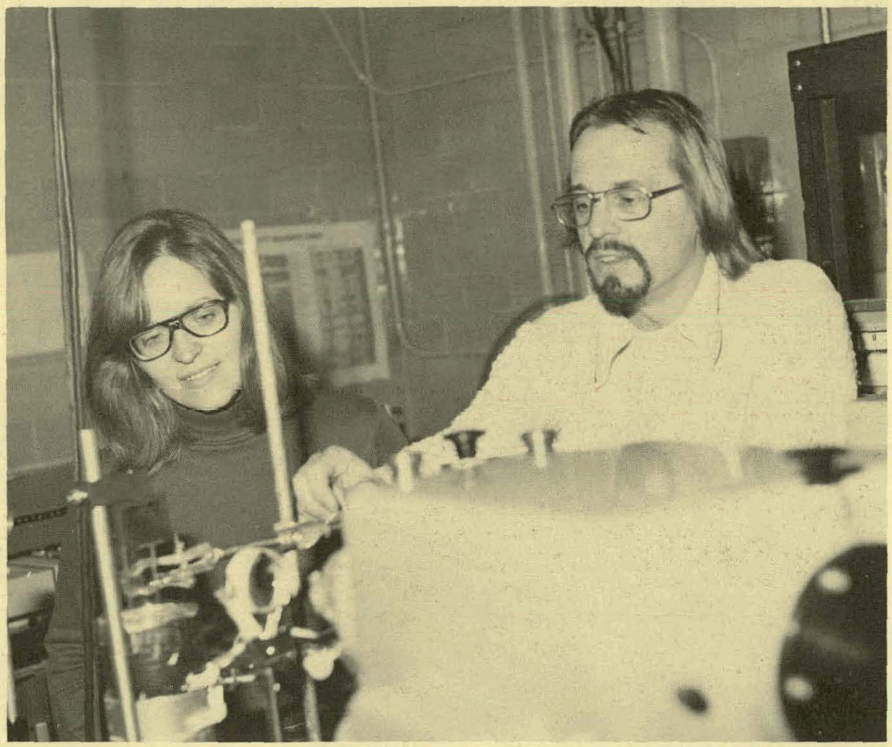


NMR spectrum of carbon 13 in coal; use of combined cross polarization, strong hetero-nuclear decoupling, and magic angle spinning to distinguish aliphatic from aromatic carbons.

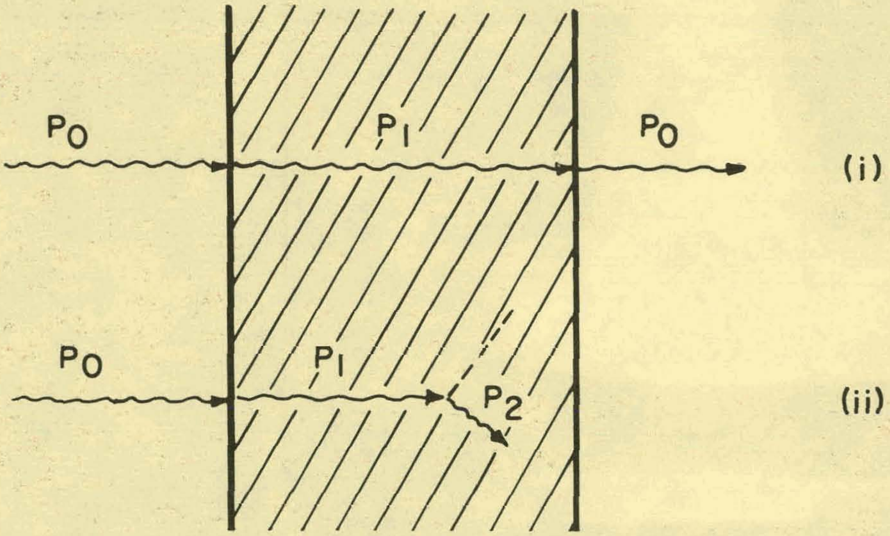
Perfect Molecular Crystals

For the first time in this country, Dr. Gerald J. Small, chemist, and Susan Robinette, graduate assistant, have positively confirmed the Hopfield theory for the mechanism of light wave transmission in crystals. In the late 1950's, J. J. Hopfield envisioned light absorption as a two-step process, the second step depending on the excited state of interest evolving into other states of the crystal. This evolution will not occur in a perfect crystal and, consequently, there can be no absorption. (See accompanying diagram.)

Small and Robinette examined all of the conditions for perfect crystal fabrication: absolutely identical, repeating molecular units in three-dimensions; and crystals that are strain-free, vibrationless, and free of defects such as chemical impurities. They found that under proper conditions, naphthalene (common moth crystals) forms nearly perfect crystals. Increasing the crystal's purity and lowering the temperature resulted in less light absorption and a more "perfect" crystal. This knowledge aids in understanding the fundamental light absorption principle for all crystals.



Susan Robinette and Gerald Small are shown with an optical spectrometer that is used in light absorption studies.



Mechanism for transmission of a photon in (i) perfect crystal, (ii) imperfect crystal. P_0 = photon; P_1 = polariton; P_2 = scattered polariton.

Luminous vapors from Velmer Fassel's emission spectroscopic work.

Beauty, Truth and Life

by Klaus Ruedenberg

Scientists at the Ames Laboratory are aware of and sensitive to the beauty of nature as they find it in their endeavors, particularly on the atomic scale. In a lecture on Science, Art and Beauty at the Design Center of Iowa State University, the author projected a scientist's view of the relationship between art and science.

The art-appreciative audience of painters, sculptors, designers, architects and other connoisseurs were surprised and excited by the about sixty slides that accompanied the lecture. They originated from various research groups in the Ames Laboratory. Besides his own computer drawn plots of electron waves in molecules, the speaker showed x-ray structures determined by Drs. Robert A. Jacobson and Jon C. Clardy, field emission microscopic photographs of absorbates on tungsten metal tips from Dr. Robert S. Hansen's surface investigations, crystallized salts from Dr. Bernard C. Gerstein, computer drawings from Dr. John D. Corbett's molecular structure studies, luminous vapors from Dr. Velmer A. Fassel's emission spectroscopic work and photographs of thin solid slices under polarized light from the metallurgical work of Drs. David T. Peterson, John D. Verhoeven and Mr. Harlan H. Baker.

Attempting to find a common ground for artists and scientists, the author developed in his lecture the following ideas.

Art is an effort to communicate human experience by means of concrete forms which differ according to the physical medium of the artistic field, such as music, visual arts, and literature. The aim of artistic activity may be the enjoyment and edification of the creator as well as the beholder, or it may be purification and catharsis. Always, however, it is a deepening of our life senses. Art testifies to the underlying promise of life, it holds out hope in the powers of growth, unfolding and healing in life. It proclaims trust in the constructive aspects of creativity.

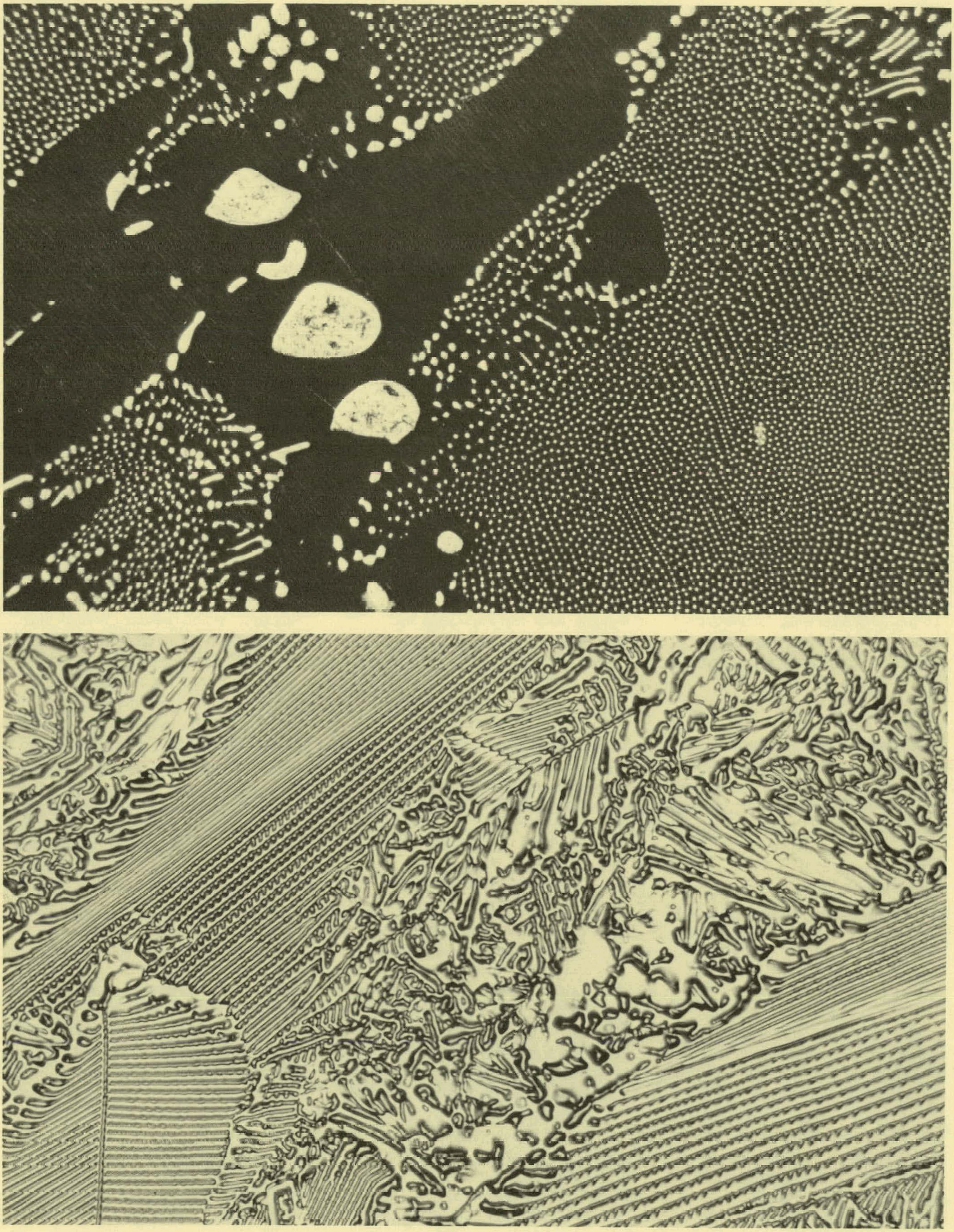
This underlying aim is essentially conveyed by means of the beauty and the quality of the *form* whose creation is the object of the artist's efforts. It is particularly poignant when the artist focuses his attention on the destructive aspects of human activity, such as war, poverty, discrimination, and hate. In such cases, there exists a dramatic tension between the depicted human failure and the quality of the artistic form. It is the latter which implies that there exist stronger and deeper forces that work *for* life. In many cases, in the speaker's opinion probably in all cases, it is only through the quality of form and the purity of beauty that art can honestly hold out this hope in a convincing manner.

The artist is able to do so by appealing to human resources which lie deeply buried in man's subconscious. Creativity surfaces in the play of children, but is much repressed in the games of adults. The elemental forces of life are tied to the strong emotions of pleasure and pain and therefore often are hidden by fear. The true artist strikes fire from such rock bottom.

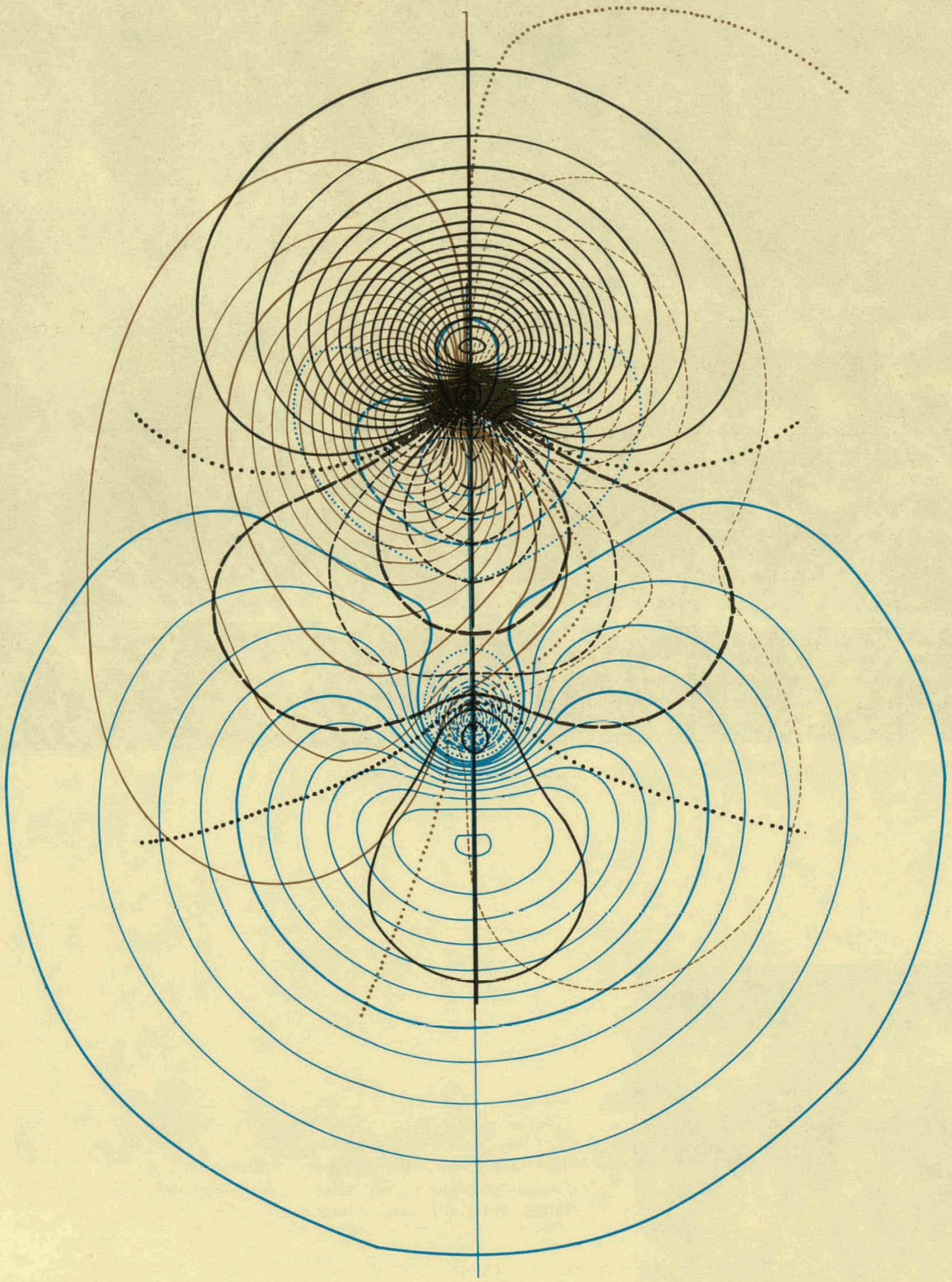
These most vigorous and violent elements of our mental activity are, however, firmly rooted in the biological base layer of our nervous system. It is therefore only natural that those forms which convey the most intense artistic and human emotions are, at heart, biological forms. Biological forms are, however, essentially physicochemical in nature, and their visual and acoustical shapes are those of specific mathematical functional relationships. Harmonies in music are related to rational proportions. The shapes of the human body, that inspire the sculptor, derive from hydrostatic pressure equilibria inside the skin membrane whose elasticity depends on biochemical equilibria.

It is, therefore, not surprising that a search among the shapes, colors and sounds occurring in chemistry, physics and biology will reveal many forms which are close to those sought by the artist. It is particularly remarkable, however, that there exists resonance between the abstract forms of modern fine arts and those of atomic and molecular phenomena. (Nearly all the artists in the audience volunteered the startled opinion that they would have taken many of the slides shown as works of modern artists, had they not been told otherwise).

The scientist who is truly in touch with nature as well as with himself, cannot help being captivated by the constructive ingenuity through which the whole of nature as well as its minute details have been and still are evolving. In the final analysis, he will have to read nature's shapes, forms and sounds in the same light as the artist: as a message of the hidden promise of life which is still embedded in nature as we know it. He will therefore tell his fellow men not only about his excitement of finding truth and understanding, not only about possible practical applications for practical human use, but also about the essential and fundamental need for preserving and caring for the fabric of nature around us if we wish to survive as humans.

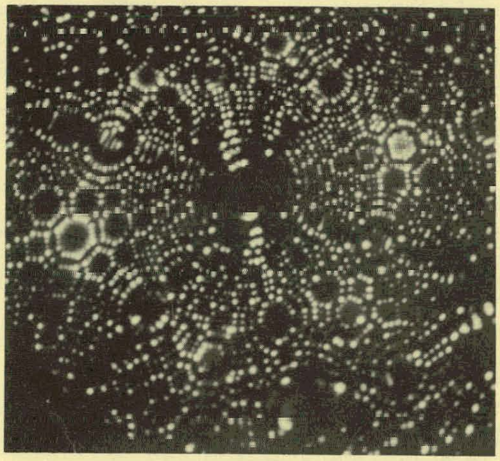


Photographs of thin solid slices under polarized light from the metallurgical work of David T. Peterson, John D. Verhoeven and Harlan H. Baker.

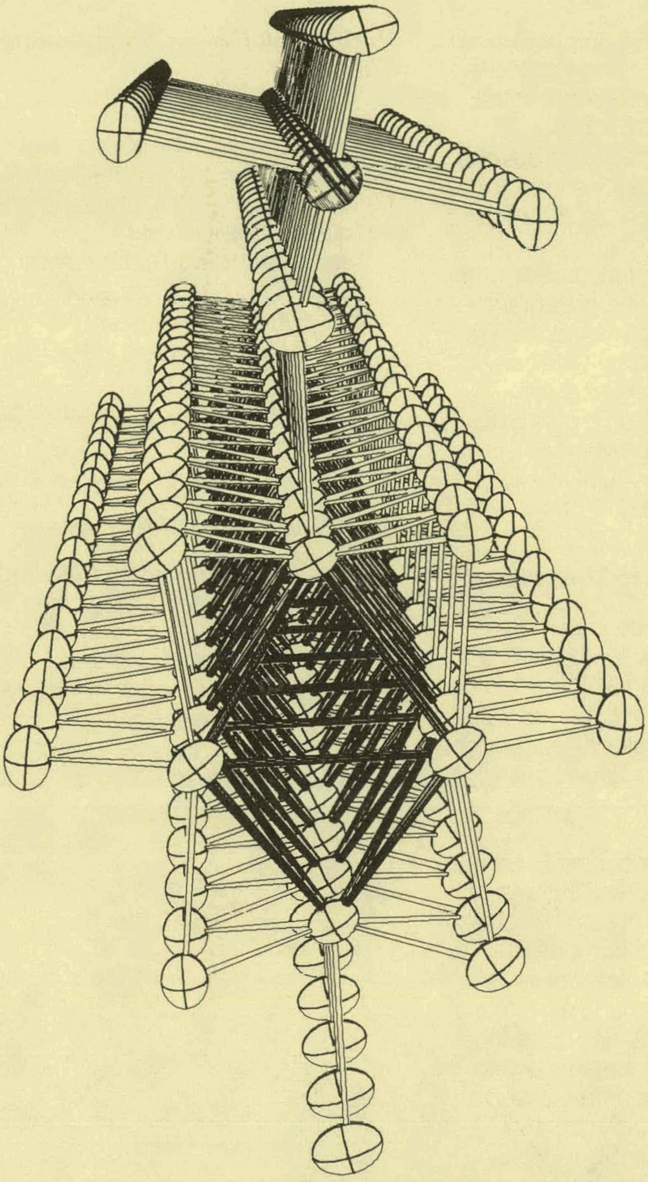


Computer drawn plots of electron waves in molecules, contributed by Klaus Ruedenberg.

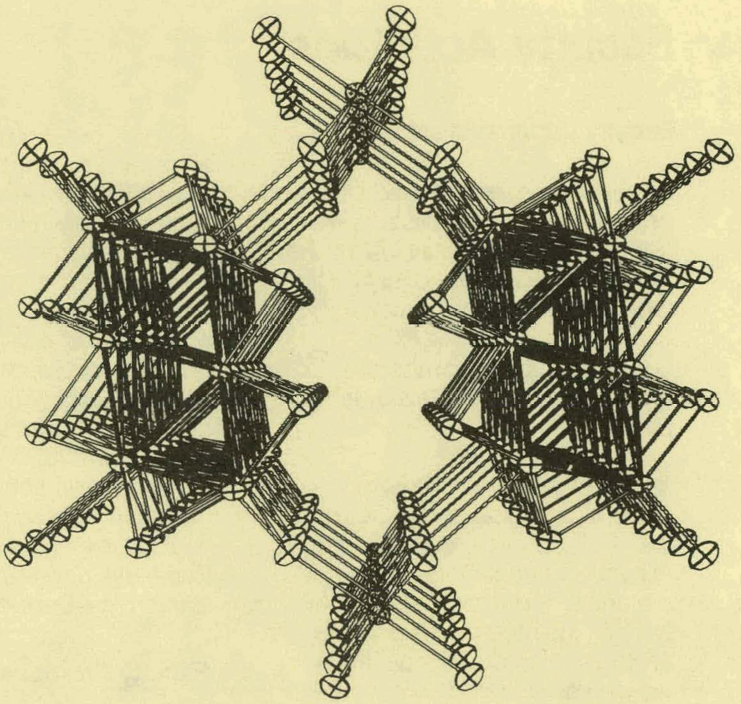
Photograph of crystallized salts ($CsCl\text{-}CuCl_2\text{-}H_2O$), contributed by Bernard C. Gerstein.



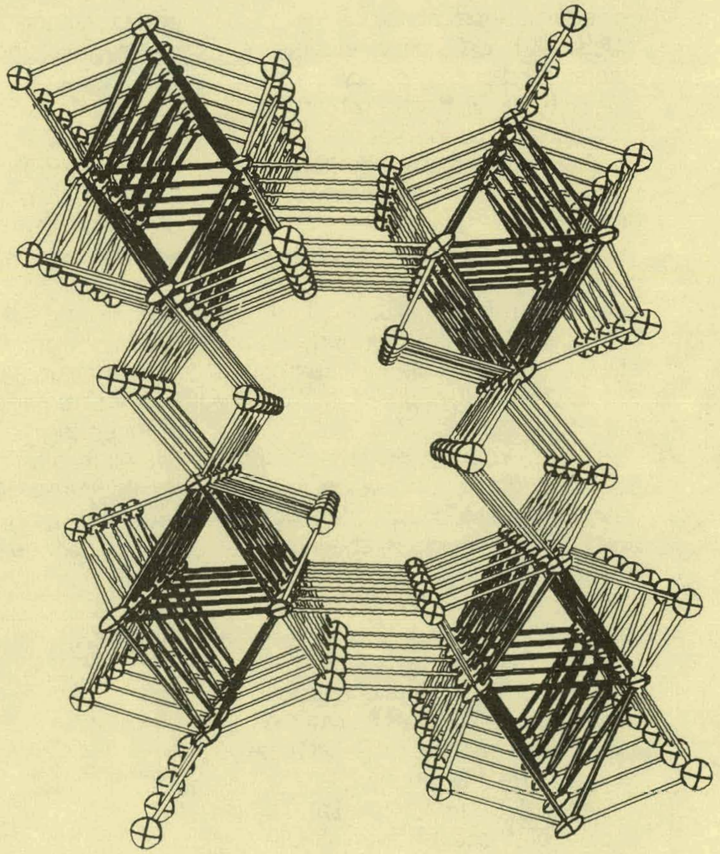
Field emission microscopic photograph of absorbates on tungsten metal tips from Robert S. Hansen's surface investigations.



$(\mathrm{ScCl_2^+})(\mathrm{Sc_4Cl_6^-})$



$(\mathrm{ScCl_2^+})(\mathrm{Sc_6Cl_8^-})$



$\mathrm{Gd_4Cl_6}$

Computer drawn representation of several scandium and gadolinium structures, contributed by John D. Corbett.

Faculty Activities

Faculty Improvement Leaves

Faculty improvement leaves for nine Ames Laboratory professors were approved in 1976 and 1977 by the Iowa State Board of Regents. The faculty improvement leave program was authorized by the Iowa Legislature to encourage professional growth of the academic staff.

Those receiving leaves in 1976 included: Michael F. Berard, ceramic engineer and professor of materials science and engineering, to be a visiting scholar in the materials department at UCLA, to work on the electronics division staff of Rockwell International, and to work on developing ceramic fabrication processes.

Laurent Hodges, physicist and associate professor of physics, to assist the congressional committee on science and technology in Washington, D.C. in formulating science and energy policy.

John F. Smith, section chief and professor of materials science and engineering, to study metallurgical thermodynamics in Aachen, West Germany.

Physicist and associate professor of physics Fred K. Wohn, to serve as a visiting professor at Justus Leibig University, Giessen, Germany, and at the Center of Nuclear Research, Service Pile Universitaire, Strasbourg, France.

Bing-Lin Young, physicist and associate professor of physics, to do research and studying at American and European nuclear research laboratories.

Those receiving leaves in 1977 included:

Robert E. McCarley, senior chemist, to investigate construction of new solid state materials at the Max Planck Institute for Solid State Science, West Germany.

Gerald J. Small, chemist and associate professor of chemistry, to work at the University of Stuttgart, West Germany, on developing an understanding of light absorption and biologically related energy transfer processes in crystals.

Senior metallurgist and professor of materials science and engineering, Rohit K. Trivedi, to work at the Technical University of Braunschweig, West Germany, to develop a theoretical model of the role of interface processes on the growth and dissolution of crystals.

Monroe S. Wechsler, section chief and professor of materials science and engineering, to work at the Max Planck Institute in West Germany studying radiation damage of metals, and to work on a new type of heat engine at the Lawrence Berkeley Laboratory, University of California—Berkeley.

Faculty Citations to Three Here

Three Ames Laboratory staff members received Iowa State University Faculty Citations in 1976 and 1977.

Premo Chiotti, senior metallurgist and professor of materials science and engineering, received a 1976 Faculty Citation. He has worked with the Lab since 1949.

Clair G. Maple and John F. Smith received 1977 Faculty Citations. Maple, assistant program director of mathematics and computer sciences, is also director of ISU's Computation Center and a professor of computer science. He joined the ISU faculty in 1950. Smith, section chief, physical properties, and professor of materials science and engineering, joined ISU in 1948.

Faculty citations are awarded by the ISU Alumni Association, and recognize long, outstanding, and inspiring service on the University faculty.

Verhoeven Named Outstanding Teacher

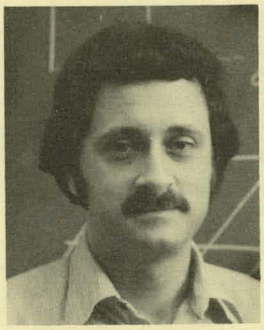
John D. Verhoeven, senior metallurgist, was one of five Iowa State University faculty members named outstanding teachers for the 1975-76 academic year.

The Outstanding Teacher Award includes a plaque and a $500 honorarium. Recipients are selected by committees in the ISU colleges.

Verhoeven joined Ames Lab and the ISU faculty in 1963.

Teaching Award to Kayser

Francis X. Kayser, metallurgist and associate professor of materials science and engineering, was one of three recipients of Superior Engineering Teaching Awards at a College of Engineering faculty convocation in 1977. Kayser joined Ames Laboratory in 1963.


Michael F. Berard

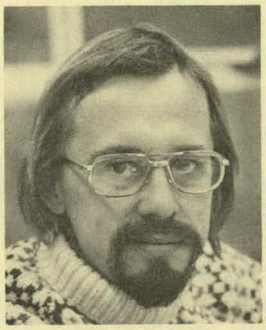

Premo Chiotti


Laurent Hodges


Francis X. Kayser


Robert E. McCarley

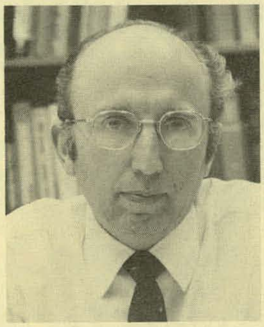

Clair G. Maple


Gerald J. Small


John F. Smith


Rohit K. Trivedi


John D. Verhoeven


Monroe S. Wechsler


Fred K. Wohn


Bing-Lin Young

Recognition

Honorary Doctorate to Ruedenberg

Klaus Ruedenberg, senior chemist, received the Honorary Doctor of Philosophy from the University of Basel, Switzerland, in 1976. The degree was conferred on Ruedenberg for his "fundamental contributions to modern quantum chemistry." While in Europe to receive the degree, Ruedenberg gave technical lectures to the Basel Chemical Society and to the Universities of Basel, Strasbourg, and Zurich.

Distinguished Professorships to Four Here

Three Ames Laboratory scientists and an Iowa Coal Project (ICP) leader were among the nine recipients of Iowa State University Distinguished Professorships awarded during 1976 and 1977.

V. A. Fassel, deputy director of Ames Laboratory, and Clayton A. Swenson, senior physicist and chairman of the Physics Department, in 1976 were named Distinguished Professors in Sciences and Humanities. Fassel joined Ames Lab in 1942 and Swenson in 1955.

The 1976 Charles F. Curtiss Distinguished Professorship in Agriculture was awarded to Neil E. Harl, ICP project leader and professor of economics.

In 1977, a Distinguished Professorship was awarded to Sam Legvold, senior physicist and professor of physics, who joined Ames Lab in 1947. Legvold was named a distinguished professor in sciences and humanities.

Distinguished Professorships are awarded by the Iowa State University Achievement Fund on the basis of faculty recommendations. The title recognizes teaching and research activities and is accompanied by a $500 honorarium. Recipients carry the title for the rest of their careers.

V. A. Fassel

Neil E. Harl

Sam Legvold

Klaus Ruedenberg

Clayton A. Swenson

Editorial, Lecture and Awards Posts to Karl Gschneidner

Karl A. Gschneidner, assistant program director, metallurgy and ceramics, in 1976 was appointed to the Board of Editorial Associates of *Semiconductors and Insulators,* a new journal of ionic and covalent solids. He served previously on the editorial board of the *Journal of Nonmetals and Semiconductors,* the forerunner of the new journal. Editorial associates can accept qualifying manuscripts for direct publication without further review.

Gschneidner was also appointed in 1977 to serve as a member of the Institute of Metals Lecture and Robert F. Mehl Award Committee of the Metallurgical Society of AIME (American Institute of Mining, Metallurgical and Petroleum Engineers) for a four year term.

Burnet is AIChE Fellow

George Burnet, senior chemical engineer and head, Department of Chemical Engineering and Nuclear Engineering, was elected a Fellow of the American Institute of Chemical Engineers (AIChE) in 1976.

Burnet is a member of the AIChE's Iowa section, and a former chairman of the American Chemical Society's division of fertilizer and soil chemistry. He joined Ames Laboratory in 1956.

Wildman to AUA Board

Ruth Wildman, Lab associate and assistant dean of sciences and humanities, was elected to the Board of Trustees of the Argonne University Association for a three-year term in 1977. The AUA, sponsored by 30 member universities including Iowa State, fosters scientific research in the Argonne National Laboratory. Wildman, as a representative in the field of biology, was assigned to the Center for Educational Affairs Committee and the Radiological and Environmental Research Committee.

Hansen to Iowa Science Council

Robert S. Hansen, Ames Laboratory director, was appointed by Iowa Governor Robert Ray to the energy panel of the 12-member Governor's Science Advisory Council in 1977. The council is to provide scientific and technological advice to the governor and to state agencies in energy, environmental quality, and natural resource areas.

Division Chiefs Appointed

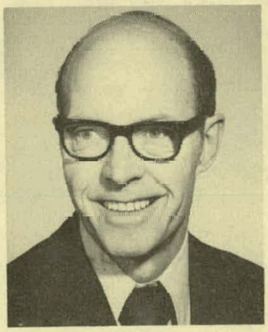
Two new division chiefs were appointed in 1977 by Ames Laboratory Director Robert S. Hansen. They are Ray W. Fisher, chief, Fossil Energy Division, and Kenneth L. Kliewer, chief, Solar Energy Division.

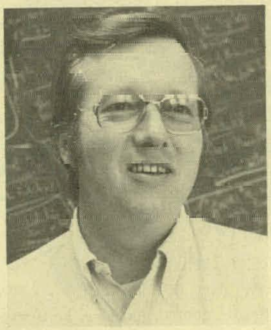
During 1977, Fisher was charged with management of programs funded through the DOE Division of Fossil Energy, and Kliewer had corresponding management of solar energy programs. Fisher was also manager, Building and Engineering Services, within the Laboratory. Kliewer was the Laboratory's assistant program director for Solid State Physics.


George Burnet


Ray W. Fisher


Karl A. Gschneidner


Robert S. Hansen


Kenneth L. Kliewer


Ruth Wildman

Laboratory Developments

Plant Protection Monitoring System

Del Bluhm (standing) gives Rudy Klingseis, plant safety sergeant, some encouragement as he familiarizes himself with the plant monitoring system.

A mini-computer, a teletype console, and two visual displays which resemble large TV sets make up the heart of the Plant Protection Monitoring System (PPMS) installed at the Ames Laboratory in 1976.

The system is the first within a Department of Energy facility to combine fire surveillance with physical security and environmental monitoring. It is also the first electronic state-of-the-art facility in the United States to meet the requirements and intents of all applicable fire codes.

Designers of the system were Delwyn Bluhm, head, plant engineering, and Wayne Rhinehart, formerly chief design engineer at the Laboratory.

Phase One of the PPMS is now in operation, and monitors nearly 300 points in the Lab complex by means of remote sensors. Included are Lab buildings, the nuclear reactor site, and Iowa State University buildings in which the Laboratory rents space.

If a sensor such as a smoke detector is activated, the PPMS operator is notified by a printed message on the cathode ray tube display, and by a graphics display which shows a floor plan with the location of the detector. The operator acknowledges the message and can request further information.

The PPMS has several backup systems and self-checking devices to ensure that transmitted information is accurate.

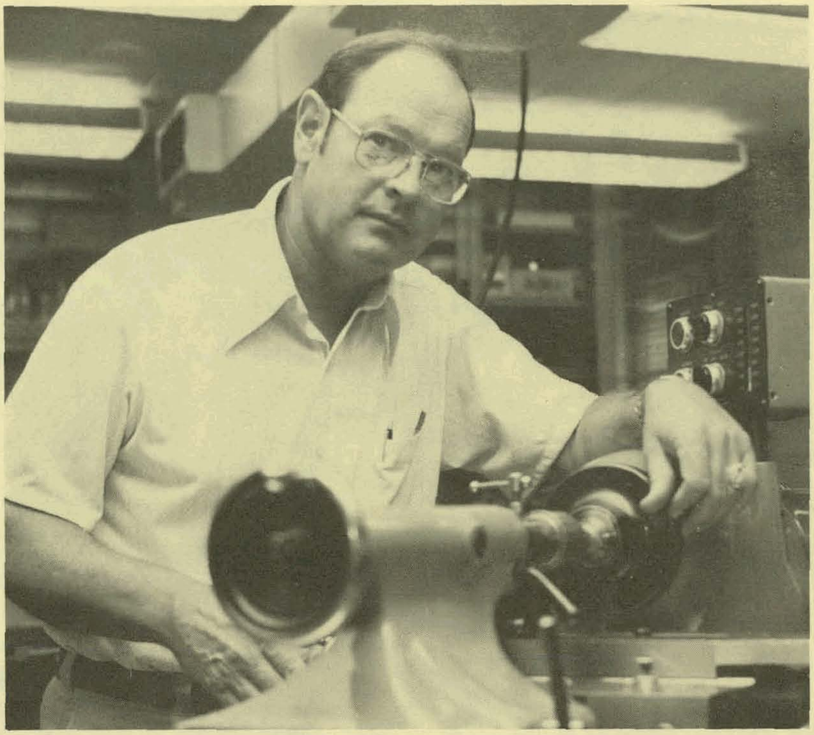
Patrol rounds are programmed into the mini-computer, and if tour stations are not punched by a walking patrolman at correct intervals, the PPMS notifies the supervisor. Radio communication between patrolmen and their supervisor is also maintained.

Phase Two of the PPMS will provide environmental monitoring and control of existing heating and ventilating systems.

Power demands in each Lab building will be continuously monitored, so that the Laboratory can take steps to reduce power consumption during peak load times. Since utility rates are based on peak demand, the new system will lower total energy costs. Phase Two is scheduled for completion in 1979.

The plan for Phase Three involves monitoring and control of experimental equipment for Lab research groups. These functions will be added to the system as funding becomes available.

The Ames Lab PPMS culminates an effort to update a 20-year-old system which performed only a few of the functions performed now. Installation of the system was done by Building and Engineering Services and Instrumentation Services personnel. System hardware was supplied by Johnson Controls, Inc., Milwaukee, Wisconsin, which built the system to Ames Laboratory specifications.



Garry Wells

Machining Precision Washers

A new punch and die method for making precision washers was developed in 1976 by Garry Wells, manager, Machine Shop Services. The simple, inexpensive process consists of machining a made-to-order punch and die out of available stock, brass or steel, for whatever size washer is desired.

Descriptions of the method have been published in *Machine and Tool Blue Book, Manufacturing Engineering, Cutting Tool Engineering,* and *Industry Week.*

Patents

Bernard Gerstein, senior chemist, and David Macaulay, formerly a junior chemist at the Laboratory, received a patent in 1976 for a method of removing carbon monoxide from gases.

The method involves passing an atmosphere containing carbon monoxide through a bed of a terbium oxide catalyst, which oxidizes the carbon monoxide to carbon dioxide.

Arthur P. D'Silva, associate chemist, and Velmer A. Fassel, deputy director, were awarded a patent in 1976 on their improved x-ray image intensifier phosphor.

Under x-ray irradiation, the yttrium-gadolinium-terbium phosphate phosphor emits intense violet-blue light in the wavelength where x-ray film used in medicine and industry is most sensitive.

Widespread use of the phosphor could result in a 10-20% reduction in radiation exposure for people receiving diagnostic medical and dental x-rays.

Premo Chiotti, senior metallurgist, and Mahesh Chandra Jha, Arvada, Colorado, were granted a patent in 1977 for a new method of separating uranium from mixed oxides of thorium and uranium.

The Chiotti-Chandra Jha process employs a molten chloride salt containing thorium tetrachloride and thorium metal, which reduces the uranium oxide to uranium metal.

John D. Verhoeven, senior metallurgist, and Edwin D. Gibson, associate metallurgist, were awarded a patent in 1977 on their techniques for producing and machining the cathodes of electron guns used in the scanning electron microscope (SEM).

The Verhoeven-Gibson process creates high-purity single-crystal cathodes of lanthanum hexaboride, which produce electron beams 15 to 20 times brighter than those of tungsten cathodes. The new cathodes are more reliable over time than conventionally-produced LaB_6 cathodes.

Verhoeven and Gibson also modified the electron gun so the cathode can be easily removed for resharpening, and they developed a simple device for grinding the tips.

1977 Group Pictures


Scientific Group


Administrative and Service Group

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