Quarterly Report
April 1, 1982 - June 30, 1982

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Prepared for
Pollutant Characterization
and Safety Research Division

Office of Health and Environmental Research
Office of Energy Research
Department of Energy

Date Transmitted: November 1982

Ames Laboratory
Iowa State University
Ames, Iowa 50011

Prepared For
The U. S. Department of Energy
Under Contract W-7405-eng -82

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by the

AMES LABORATORY, DOE
IOWA STATE UNIVERSITY
AMES, IOWA

Date Transmitted: November 1982

V. A. Fassel

Program Director

G. A. Junk

R. N. Kniseley

Assistant Program Directors

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I. Health Effects Research (HA-02-02)

1. Health and Environmental Impact of Waste and Biomass to Energy Processes (A. D'Silva, V. Fassel)

A. Progress (S. Walters)

The determination of the effects of air particulates and extracts of air particulates on organ cultures of the respiratory system is a new project. The first priority of this investigation has been a review of the literature for ideas concerning a suitable organ culture system. Trachea have the greatest exposure to airborne particulates and are susceptible to induced injury and disease. Trachea organ cultures are also used frequently in biochemical studies of the respiratory system and they are easily maintained in culture. For these reasons, they will be used in our preliminary investigations.

Organ cultures of rat trachea and lung explants have been maintained for a two week period. Histological examination of the trachea explants will be used to confirm whether normal cell types remain in culture or if they exhibit abnormalities such as changed morphology or transformation to cancer cells.

Biochemical markers of toxic and neoplastic changes will be the major indicators used in this study of the effects of air samples on trachea organ cultures. The protein and PNA content of trachea organ cultures exposed to air sample particulates and to particulate extracts will be determined as an indication of any gross abnormalities in synthesis and growth. Increased DNA in mouse lung tissue after carcinogen exposure and prior to tumor formation has been reported by H. K. DeMunter, L. Den Engelse, and P. Emmelot [Chem-Biol Interactions 24: 299-316 (1979)]. We plan to investigate changes in synthesis of specific proteins by polyacrylamide gel electrophoresis techniques. Subcellular fractionation and separation by cell type will probably be necessary.

Enzyme activities in lungs may also be altered by exposure to carcinogens as reported by T. Suzuki [GANN 57:169-184 (1966)] and P. Rady, et al. [Chem-Biol Interactions 31:209-213 (1980)]. Enzyme assays of the trachea organ cultures, or their homogenates, may thus provide good indicators of exposure to toxic compounds present on air particles and in extracts of these particles.

B. Status

Necessary equipment and supplies are still being procured. Establishing and maintaining explant cultures will be accomplished as soon as these arrive. PNA and protein assays of the trachea organ cultures should be completed shortly thereafter.

II. Physical and Technological Studies (HA-02-04)

1. Characterization of Organic Pollutants (G. Junk)

A. Progress

The feasibility of using n-dodecane (n-C₁₂) as an exchange solvent in the analyses of polycyclic aromatic hydrocarbons (PAHs) was investigated. The investigations involved chromatography, losses during evaporation, solvent volume accuracy and detection limits.

Chromatographic conditions were established to separate benz[a]pyrene from benz[e]pyrene. The capillary column was SE54 and the program was 175°C for 4 minutes and then 2°C/min to 275°C. The elevated initial column temperature was possible because of the high 200°C boiling point of the n-C₁₂ solvent. Benefits of this new chromatographic approach are reduced cycle time between analyses and a better solvent effect in the splitless injection.

A recommended method of PAHs analyses is evaporation of a solvent such as benzene to dryness and then redissolving the PAH residue in a different solvent. We have observed losses of about 30% for phenanthrene when this approach is used. However, no phenanthrene is lost when the n-C₁₂ is added to the benzene extract prior to removal of the benzene by distillation.

An additional advantage of adding the exchange solvent prior to removal of the benzene is the precision inherent in adding a small amount of n-C₁₂ to the benzene solution. For example, as little as 10 µl of n-C₁₂ can be added and completely retained during the benzene evaporation step.

Absolute detection limits for PAHs using this transfer to n-C₁₂ are comparable or slightly better than those obtained previously.

Some additional investigations of extraction procedures were accomplished during this quarter. When the basic fly ash taken from the electrostatic precipitator of coal-fired power plant was pretreated with aqueous HCl, the amount of carboxylic acids that could be extracted was doubled. Tests of the extraction procedure involved benzene as the extracting solvent. For the carboxylic acids, the more simple and rapid probe sonication was just as effective as the soxhlet extraction of the fly ash.

The laboratory validation studies of the Ames vapor sampling system (AVSS) were completed. Eighty-five different compounds comprising twelve different chemical classes were tested at simulated concentrations ranging from 0.01 to 10 ng per liter. The average percent recoveries ranged from 76% for esters to 100% for pesticides. The overall weighted average percent recovery was an excellent 91%.

B. Status

A report describing the design and operation of a simple effective sampler, based on the use of the synthetic polymer XAD-2, will be prepared for publication as a chapter in an environmental book to be edited by L. H. Keith and published by Ann Arbor Science. This report will include the design and fabrication of the sampler and the results of laboratory and field tests. Several applications will also be included to demonstrate the usefulness of the sampler in solving environmental problems.

2. Environmental Effects of Using Municipal Solid Waste as a Supplementary Fuel (G. Junk)

ORGANIC ANALYSES

A. Progress

Fly ash taken from the electrostatic precipitator at the Ames power plant was investigated for the presence of acidic material. Carboxylic acids were the dominant group of acidic components isolated from the fly ash and separated and detected by gas chromatography. Acids from C₁₀ through C₁₈ were observed in concentrations from 0.01 µg/g for decanoic acid to 0.27 µg/g for palmitic acid. Sonication and soxhlet extractions of the fly ash, with and without HCl acid pretreatments, were employed. In all cases, higher amounts of the organic acids were extracted when the basic fly ash was pretreated with aqueous HCl.

The treated fly ash was also extracted and analyzed for polycyclic aromatic hydrocarbons. None were found at a detection limit of one ppB or one ng/g of fly ash. These results confirm all the previous analyses of different samples taken from the Ames power plant.

B. Status

All of the organic analytical data for the investigation of the environmental aspects of co-combusting mixtures of coal and processed municipal refuse are being organized and collated for inclusion in a final report. The data from one aspect of this final report have already been organized and these data were discussed in an invited paper presented at the Conference on Resource Recovery from Municipal, Hazardous and Coal Solid Wastes held in Miami, May, 1982. The title of the paper was "Coal/Refuse Combustions: 1. Organic Emissions". A similar report on "Inorganic Emissions" was presented by G. Norton.

INORGANIC ANALYSES (G. Norton)

A. Progress

Collation of SO_x, NO_x, and total suspended particulate (TSP) emission data from Ames and other coal/RDF sites has been completed and a preliminary summary and comparison of these data have been prepared. In general, emission trends for the regulated pollutants at Ames followed the same trends observed at other sites. Generally, SO_x emissions substantially decreased and NO_x emissions slightly decreased at all sites when RDF was fired with coal. The effect of co-combustion on TSP emissions was variable.

Bottom ash, electrostatic precipitator (ESP) fly ash, and sized suspended stack particulates from the tangentially-fired unit at Ames were analyzed for major, minor, and trace elements by neutron activation, x-ray fluorescence, inductively coupled

plasma-atomic emission spectroscopy, and atomic absorption. In order to provide the chemical composition of unsized stack particulates, analytical data on the separate size fractions were used to obtain a weighted average.

In the fly ash, none of the determined constituents increased by a factor of two or more as a result of co-combustion. Levels of Br, Cr, Na, Ni, Pb, Sb, and Zn increased two to four times in the bottom ash. Weighted averages of the stack ash showed only cadmium to increase by at least a factor of two. However, in the highly respirable 1 to 3 micron size fraction, Cd, Pb, Sb, and Zn increased by factors of two to four. Analysis of the submicron fraction as a function of particle size has not yet been completed. The overlap in elements determined by the different analytical methods will provide a good set of quantitative data for these samples.

B. Status

All analytical determinations for inorganic constituents have now been completed. With the collation of the regulated pollutant data now essentially completed, emphasis has shifted towards collating the remainder of the inorganic data base, particularly with respect to data on sized suspended stack particulate matter obtained with the Source Assessment Sampling System. Progress continues to be made towards the formulation of a comprehensive report of the Ames study, including an interstudy comparison of emission results from other coal and coal/RDF combustion facilities.

3. Microbiological Air Quality of the Ames Municipal Solid Waste Recovery System (R. Kniseley and L. Lembke)

A. Progress

Dr. Merlin Bicking completed his Ph.D. dissertation, "New Analytical Techniques for Mycotoxins in Complex Organic Matrices", and it was accepted by the Graduate College at Iowa State University on May 5, 1982. Drafts for three publications based on his work have been prepared.

A paper titled "Microbiological Air Quality in the Ames Solid Waste Recovery System" was prepared and presented at the Conference on Resource Recovery from Municipal, Hazardous and Coal Solid Wastes held in Miami, May, 1982.

Work on summarizing the data obtained from this study has continued in order to prepare a final report. Draft versions of several sections of this report have been prepared.

B. Status

Major efforts will be directed toward completion of publications describing the results of the microbial studies at the Ames solid waste facility. It is anticipated that four publications will result in addition to the three papers that have been published. A final project report will also be completed.

4. Solid Waste to Methane Study (C. Chriswell)

A. Progress

Hydrogen sulfide can be recovered quantitatively from digester gas and from other gaseous streams by adsorption on a molecular sieve. The procedure is effective either for accumulating trace quantities of hydrogen sulfide as a prelude to determinations or for removing hydrogen sulfide from gases. However, effective procedures already exist for determining and for scrubbing hydrogen sulfide from gaseous streams. The apparent need to accumulate hydrogen sulfide from hot gaseous streams in fuel cells is an application of the molecular sieve that is being investigated currently.

Preliminary investigations have been completed on several novel techniques for recovering organic components from particles. Previous work has shown that treatment of particles with hydrofluoric acid leads to enhanced recovery of some organic components. It has been found that treatment with hydrogen fluoride is effective, rapid, and leaves the treated ash in a form more suitable for subsequent extractions. However, these benefits are balanced against the inconvenience involved in using a highly corrosive gas. Further work is required to determine if a more safe and convenient procedure can be developed.

Glacial acetic acid as free of water as possible also appears to be an effective agent for the pretreatment of particles. Further work will be performed using acetic acid-acetic anhydride mixtures as a pretreatment agent.

Preliminary investigations have begun on the potential use of high boiling point compounds as extraction solvents for particles. Studies by Walters and D'Silva have already shown that the use of triphenylmethane and diphenylether can lead to enhanced recovery of PAHs from fly ash. In the present work, liquid phases used in gas chromatography are being studied. These compounds are available in high purity and their boiling points and affinity for organic components are ideal for extraction purposes. However many of these compounds will not wet fly ash. Carbowax 20M and 1,10-phenanthroline may be suitable for use as extraction agents. The 1,10-phenanthroline is a very powerful complexing agent and also leads to the extraction of metals from the fly ash. If further investigations show this property is undesirable, a compound such as 4,7-phenanthroline, which should not be a good complexing agent, will be studied.

B. Status

The solid waste to methane environmental study will end this fiscal year and ongoing work will be performed under other projects. One of these ongoing projects is the investigation of silicalite for environmental applications. A report describing the use of this molecular sieve for accumulating sulfur dioxide from stack gases has already been published in Analytical Chemistry. A reprint is attached as Appendix A.

5. High Resolution Luminescence Spectroscopy

5a. X-ray and Laser Excited Shpol'skii Spectroscopy (A. D'Silva, V. Fassel)

A. Progress (Y. Yang)

The quasiline luminescence spectrum of 3-amino pyrene has been observed in the LESS system that attains temperatures in the 2 to 4K range. Absorption spectra of aromatic amines in n-alkane solvents indicate that charge transfer complexes are not formed, as has been suggested to occur in glass forming solvents. Whether such an observation is valid for a wide variety of aromatic amines has yet to be evaluated. Extremely sharp line luminescence spectra have also been observed for the compound 2,3,2',5'-tetrachlorobiphenyl in n-hexane at 4K, which is an indication that the LESS technique has the potential to detect halogenated aromatics such as dioxins.

A copy of the manuscript entitled "Characterization of an Immobilized Enzyme System for the Conversion of Benzo[a]Pyrene to Fluorescent Metabolites", which has been submitted to Analytical Chemistry for publication, is included as Appendix B to this report.

B. Status

The characterization of a high temperature solvent extract of the atmospheric particulate matter SRM 1649 for a variety of PAHs is continuing. The system for the observation of tunable molecular ionization and molecular ion luminescence has been assembled and is being tested.

5b. Rotationally Cooled Fluorescence Spectroscopy (RCFS)
(G. Small, J. Hayes)

A. Progress

A reprint of the manuscript detailing quantitation of RCF by coupling with a simple gas chromatograph is attached as Appendix C. As noted in the previous report, the continuous flow nozzle had been out of service to test the pulsed nozzle system. Those tests have now been completed and the forepump manifold has been redesigned so that either the continuous or pulsed nozzle may be used. Some difficulty was encountered in putting the pulsed nozzle into operation, but these problems have been overcome and the system is now operating as anticipated. During the period in which the continuous nozzle was out of service, a prototype device for analysis of aqueous samples was prepared.

B. Status

Both pulsed and continuous nozzle systems are operational. During the coming quarter, the study of the spectroscopy of the aminonaphthalenes will be completed. The spectroscopy of several of the compounds of interest in air particulate analysis will be investigated and the extract of a NBS-supplied air particulate sample will be examined by RCF/GC.

5c. Fluorescence Line Narrowing Spectroscopy (FLNS)
(G. Small, J. Hayes)

A. Progress (M. McGlade)

Work on the application of FLNS to amino derivatives of polycyclic aromatic hydrocarbons was continued. The particular amines of interest are derivatives of naphthalene, phenanthrene, fluorene and fluoranthene. The $\pi \rightarrow \pi^*$ absorption transitions required for observation of FLN for these molecules all occur at wavelengths not accessible with the standard output of the N_2 -laser pumped dye laser. The laser was upgraded to include an amplifier stage and frequency doubling system. This upgrade allows either the N_2 -pumped system or the Nd:YAG pumped system to be used for this work. Problems of solubilizing the amines in an acidified glass have been overcome. Low temperature absorption spectroscopy has verified that the lower energy $n \rightarrow \pi^*$ charge transfer absorption of the amines is suppressed in the acidified glass. As previously reported, suppression of this absorption for aminoanthracenes and aminopyrenes did allow FLN spectra to be generated.

B. Status

That FLN spectra for the above compounds can be generated will be verified. Detection limits and calibration curves will then be obtained. In addition, FLNS analysis for several PAHs will be performed on the extract of an NBS-supplied air particulate sample.

6. Lead Mission - Environmental Aspects of Energy Recovery from Waste and Biomass (T. Wessels, J. O'Toole, R. Kniseley, V. Fassel, G. Junk)

A. Progress

The software for operation of the environmental document library has been developed and tested. FORTRAN programming was used to connect input files consisting of author(s), title, location, keywords, and accession number, to an indexed master list as well as indexed search files. The EDZ text editor performs character string searches of the indexed files. Additional FORTRAN programs are used to format lists of references and to unload and reload the indexed files.

B. Status

The environmental document center is operational and continues to expand through the addition of new references. Over 5,000 references have been entered into the computer index files.

Software revisions have enhanced the operation of the system.

Presently a data base management software package compatible with the DEC VAX computer is being reviewed.

III. Health and Environmental Risk Analysis (HA-02-06)

1. Risk Assessment of Municipal Wastes as a Supplemental Fuel (J. O'Toole, T. Wessels)

A. Progress

A presentation on the health and environmental effects of the energy conversion technologies in the co-combustion of solid waste was made in May, 1982 at the Conference on Resource Recovery from Municipal, Hazardous and Coal Solid Wastes, Department of Mechanical Engineering, University of Miami.

The potential uptake of toxic organic and inorganic compounds through public exposure were viewed through dispersion modeling and physiological dynamics. Potential threats of co-combustion facilities were viewed in the light of alternative disposal and energy generating options by applying existing monitoring and assessment data.

B. Status

Efforts are continuing in developing health risk assessments from the range of data on toxic emissions with particular attention to carcinogenic, genetic and epigenetic factors. The role of leachates from ash disposal sites in ground water contamination are also under evaluation. Exposure potential through site monitoring data is being assessed.

Sampling of Stack Gas for Sulfur Dioxide with a Molecular Sieve Adsorbent

Colin D. Chriswell* and Douglas T. Gjerd¹

Ames Laboratory USDOE, Iowa State University, Ames, Iowa 50011

Atmospheric sulfur dioxide has been shown to aggravate human respiratory difficulties, to damage vegetation, to accelerate the deterioration of calcareous building materials, and to be a major contributor to acid rain (1-3). The sources of this pollutant include fossil fuel combustion, cement manufacture, metal smelting, and paper pulping (1-3). Various procedures are available for determining sulfur dioxide in gaseous effluents such as stack gas. Most of these rely on the use of impinger trains to trap sulfur dioxide (4). Procedures in which sulfur dioxide is accumulated with an impinger are effective in yielding accurate and reproducible results. However, even under the best of circumstances the sampling of a stack is inconvenient, and the need to transport a liquid-filled impinger train to a sampling site on the side of a stack does little to enhance the convenience of sampling.

In the present work it has been found that a unique, hydrophobic molecular sieve can be used as an adsorbent to accumulate sulfur dioxide from stack gas. A small, pencil-sized, adsorption column replaces the impinger train normally used and, thus, greatly increases the convenience of sampling. Sulfur dioxide is quantitatively adsorbed until the capacity of the molecular sieve is exceeded. Major components of stack gas such as nitrogen, oxygen, carbon dioxide, and water vapor do not interfere with the accumulation of sulfur dioxide. Many of the trace components found in stack gas are accumulated by adsorption on Silicalite (5) and an obvious extension of the present work would be to couple the accumulation technique with thermal desorption and gas chromatography to determine other low-molecular-weight components of gaseous effluents. The adsorption properties of Silicalite also suggest potential applications for scrubbing stack gas and application has been filed for a patent on this use.

EXPERIMENTAL SECTION

Materials. The adsorbent used is a silica-based, hydrophobic molecular sieve known as Silicalite (Linde Division, Union Carbide, Tarrytown, NY). It contains 6 Å diameter pores as part of the crystal structure and these pores occupy 33% of the crystal volume (6). Various binders are used with this molecular sieve, and it has been found that they can affect the adsorption properties (5). In the present work a material designated as LZ-115 was used. This material is in the form of 20 × 80 mesh granules and is believed to contain an alumina-silicate clay binder. The molecular sieve is heated at 800 °C for 24 h to remove any adsorbed impurities and then sized to yield a 60 × 80 mesh fraction which is used for sulfur dioxide accumulation.

Synthetic stack gas (Matheson, East Rutherford, NJ) containing 543 ppm sulfur dioxide, 10.34% carbon dioxide, 9.38% oxygen, and the balance nitrogen was used during the development of the analytical protocol. Other gases used were of a purity of at least 99%.

Recommended Procedure for Determining Sulfur Dioxide. Load 4.6 mm i.d. × 5 cm stainless steel adsorption tubes with about 0.5 g of cleaned, sized Silicalite. Attach a sampling probe to the inlet of one adsorption tube, a second adsorption tube to the outlet of the first, and an air pump such as the Air Cadet (Cole Palmer, Chicago, IL) to the outlet of the second adsorption tube. The probe in this instance consists of a 3 ft length of 1/4 in. stainless steel tubing which is inserted through a sampling port with one end in the gas stream and the adsorption tubes outside the stack. Draw stack gas through the adsorption beds at a measured flow rate of approximately 100 mL/min. The total volume sampled will be determined by the sensitivity of the

Table I. Adsorption of Gases on Silicalite Molecular Sieve distribn coeff vs. He

gas	50 °C	100 °C
sulfur dioxide	1600 ^a	130
carbon dioxide	77	16
nitrous oxide	9	3
methane	24	7
nitrogen	9	3
oxygen	9	4
water	70	20
carbon monoxide	11	4
propane	1300 ^a	220
carbonyl sulfide	>1000	>500
dimethyl mercaptan	>1000	>500
hydrogen sulfide	150	40

^a Approximate.

analytical procedure used and the sulfur dioxide content of the stack gas. In the present instance a volume of 1 L was found to be adequate.

At the conclusion of sampling, transport the adsorption tubes to the laboratory, unload the molecular sieve into a combustion boat, and determine the total sulfur content using a Fisher Model 470 sulfur analyzer (Fisher Scientific, Chicago, IL) or a similar instrument. The presence of sulfur in the second adsorption tube will indicate that the capacity of the first bed was exceeded and would require resampling of a smaller volume of gas. Blank determinations should be performed on replicate samples of Silicalite not used for sampling, but the sulfur content of cleaned Silicalite and of Silicalite taken from the second adsorption bed has consistently been lower than the detection limit of the sulfur analyzer.

$$\text{ppm SO}_2 = \frac{(6.827)(\% \text{ S in Silicalite})(\text{wt of Silicalite, mg})}{(\text{volume of gas sampled, L})}$$

RESULTS AND DISCUSSION

Adsorption of Gases on Silicalite. The distribution coefficients obtained for various gases indicate that Silicalite can be used for accumulating different substances from stack gas (Table I). The distribution coefficients given were determined by gas chromatography using pure compounds

$$D_g = \frac{(\text{retention time} \times \text{flow rate}) - \text{dead volume}}{\text{wt of Silicalite}}$$

D_g has units of mL/g. Of the prevalent components of stack gas sulfur dioxide has the highest distribution coefficient and this provides the basis for its accumulation in the presence of gases such as carbon dioxide, nitrogen, oxygen, water vapor, carbon monoxide, and nitrous oxide. In the case of water vapor and carbon dioxide there was concern that mass effects due to the high concentrations of these species in stack gas would lead to the displacement of the more strongly adsorbed sulfur dioxide. However, this did not occur. Other studies (5, 7) have shown that the capacity of Silicalite for carbon dioxide and for water is relatively low even when these components are accumulated from pure streams. Thus, it appears that there is no competition for adsorption sites between sulfur dioxide and water vapor or carbon dioxide. In the case of water vapor specifically, there is strong evidence (7) that all water retained is adsorbed on the surface possibly by interactions with the binder and that no water enters the pore structure.

¹ Present address: Exxon Research and Development, Linden, NJ.

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OTHER Quarterly Report Mailing List - April, 1982

Dr. Robert W. Wood
Director
Pollutant Characterization and Safety
Research Division
Office of Health and Environmental
Research
Office of Energy Research
Department of Energy
Washington, D.C. 20545

Dr. Gerald Goldstein
Pollutant Characterization and Safety
Research Division
Office of Health & Environmental Research
Office of Energy Research
U.S. Department of Energy
Washington, D.C. 20545

Dr. Nathaniel Barr
Manager
Health and Environmental Risk Analysis
Office of Health and Environmental
Research, EV-31
Office of Energy Research
Department of Energy
Washington, D.C. 20545

Dr. Charles W. Edington
Deputy Director
Office of Health & Environmental
Research, EV-301
Office of Energy Research
U.S. Department of Energy
Washington, D.C. 20545

Dr. Martin L. Minthorn, Jr., Director
Health Effects Research Division
Office of Health & Environmental
Research, EV-33, GTN
Office of Energy Research
Department of Energy
Washington, D.C. 20545

Mr. Donald Knowles
Industrial Hygienist
Environmental Investigations Branch
NIOSH (Room 117)
944 Chestnut Ridge Road
Morgantown, W. Virginia 26505

Dr. Subrata Sengupta
Department of Mechanical Engineering
University of Miami
P.O. Box 24894
Coral Gables, Florida 33124

Mr. Bill Rosenkranz
OALWU (RD-682)
Environmental Protection Agency
401 M Street, S.W.
Washington, D.C. 20460

Dr. Robert A. Olexsey
26 West St. Clair
U.S. E.P.A.
Cincinnati, Ohio 45268

Mr. Alden Christianson/George Huffman
Solid & Hazardous Waste Research
Laboratory
Fuels Technology Branch
U.S. Environmental Protection Agency
National Environmental Research Center
Cincinnati, OH 45268

Mr. Carlton A. Wiles
Solid & Hazardous Waste Research
Laboratory
U.S. Environmental Protection Agency
National Environmental Research Center
Cincinnati, Ohio 45268

Mr. Donald K. Walter
Director
Office of Energy from Municipal Wastes
Urban Waste Technology Branch
Office of Conservation and Solar
Applications
Mail Station 2221C
Department of Energy
Washington, D.C. 20545

Dr. Roger McClelland
Inhalation Toxicology Research
Institute
Lovelace Biomedical and Environmental
Research Institute
P.O. Box 5890
Albuquerque, N.M. 87115

OHER Quarterly Report Mailing List - June 1982
(cont.)

Nancy Kaiser
Southern States Governors Conference
1 Exchange Place
Suite 1230
2300 Peachford Road
Atlanta, Georgia 30338

C. Chriswell
316 Metallurgy

E. DeKalb
8 Spedding

A. D'Silva
9 Spedding

J. Hayes
4 O & L

G. Junk
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