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REPORT OF THE

ARMY SCIENTIFIC ADVISORY PANEL

AD HOC GROUP

TRACE GAS DETECTION

NOVEMBER 1973

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I. Background:

The United States Army has always been keenly interested in finding new means of detection. For example, one of the Army's most exasperating problems has been the detection of mines. The approaches which have generally been used in mine detection are to find a unique electronic or magnetic signature of the objective which can be detected against a background of other signatures.

A second objective of detection has been to obtain early warning of chemical and biological attacks. The lethal concentrations of these materials have generally been in the parts per million level. This problem was particularly enhanced by the discovery of the fast acting nerve gases.

The Southeast Asia conflict has made the Army aware of still another detection problem: how can one detect the enemy himself in low intensity warfare? One of the main problems throughout the course of this war has been to find the enemy. Since personnel do not have characteristic electronic and magnetic signatures, it became evident that one of the means of detecting people might be through their chemical effluents. However, the concentration levels which are diffused into the atmosphere are in parts per billion or lower. Methods of chemical analysis would have to be drastically improved before these low level signatures could be successfully detected.

The first approach to this problem was the attempt to detect ammonia which is given off by the human body and which is formed in the decomposition of urine. The original device which was developed by a contractor for the Land Warfare Lab consisted of two parts. The first part of the instrument reacted the sampled atmosphere with hydrogen chloride to form particles of ammonium chloride. These particles would then act as condensation nuclei in a particulate counter. By counting these particles, "activity" could then be detected. Although simple calculations quickly showed that ammonia levels would in no case be high enough for any detection by this method, the device still seemed to detect activity. It was later discovered that the first part of the instrument could be completely removed, and "activity" was still detected. The final design of the instrument which is now called the CN detector actually measures the total particulate count over a jungle area. In areas where there is some human activity under the jungle canopy the total count of particulates in the atmosphere increases dramatically. Obviously a low background count is necessary.

Though it was first believed that the particulates originated from motion and dust being raised from the ground or directly from human effluvia, it is now accepted that the particles originate from campfires and cooking stoves which put out fine particulates in the form of smoke. Even after the smoke has been diluted to the point of being visually unobservable, it can still be detected by the CN detector. By aeri ally plotting a gradient map of the particulate count, the general area where a large accumulation of men is located can usually be determined. As may be expected there are many reasons for obtaining false signals in

such a device. Since the CN detector detects any particulate regardless of its origin, the instrument has no selectivity.

Spurred on by the apparent success of this effort, various Army laboratories have begun development of more sophisticated and sensitive means of detecting chemical effluents. It became apparent that if standard means of chemical analysis could detect parts per billion or parts per trillion of volatile components which are originating from a target then it might be possible to routinely detect explosives such as TNT and dynamite, possibly mines, bombs and concealed weapons, vehicles, drugs and narcotics, as well as personnel.

Since these techniques provided a new facet to the detection problem an ASAP study was proposed to Lieutenant General Gribble, Chief of Research and Development who approved the study. Mr. Robert L. Johnson, Assistant Secretary of the Army (Research and Development) requested that four members of ASAP serve on the Ad Hoc Group. The group consisted of Dr. Robert A. Beaudet, Professor, Department of Chemistry, University of Southern California; Dr. James J. Renier, Vice-President and General Manager, Data Systems Operations, Honeywell Inc.; Dr. Max Garbuny, Consultant, Westinghouse Company, Pittsburgh, Pennsylvania; Dr. Harold Weber, Consultant.

The problem which was given to the ASAP Ad Hoc Committee was to review the research and development in the trace gas area and assess its potential. The terms of reference are given in Appendix A of this report. The Ad Hoc Group met at approximately three different times for the purpose of being briefed by the Land Warfare Lab, The Mobility Equipment Research and Development Center, Edgewood Arsenal,

Deseret Test Center, and the Cold Regions Research Laboratory. A list of briefers is given in Appendix B. The panel was exposed to all the methods of effluent detection which are presently being studied and developed. The Ad Hoc Group has reviewed these techniques as well as the Army needs and requirements and the promise of each method.

II. Summary:

Trace gas analysis and identification of vapors is a relatively new and unexplored approach to detection. The Army has always had a deep interest in all detection techniques. It has found many applications in detection of mines, tunnels, personnel, chemical and biological agents, etc. Trace gas analysis should be considered as just another approach to solving these detection problems. Therefore, the Army should keep abreast of techniques of vapor detection and any developments in this field which might be applicable to its mission.

Since this is a relatively new field, it has been developing rapidly, spurred on by the growing interest in the monitoring of air pollution and a growing interest in the enforcement of narcotic laws.

The techniques of trace gas detection may be classified into two categories, remote sensing and chemical sniffing. Remote sensing is concerned with the real-time detection of vapors from a distance. While the older methods used adsorption spectroscopy present schemes use laser techniques. The second general approach, chemical sniffing, depends on sampling a vapor plume being emanated from a source. Subsequently, the plume is analyzed within seconds to determine what foreign volatiles are present. The former approach is obviously very attractive for the detection of air pollutants from smoke stacks, clouds of chemicals, or biological agents.

effluvia emanating from buildings, etc. Chemical sniffing seems more adaptable to detection of volatiles from close distances such as explosives, narcotics, chemical and bio agents. It is with these two techniques that this panel has concerned itself.

We have found generally that the Army has lacked a systematic approach to the problem because the techniques are new. In many cases the Army laboratories have been satisfied to "hobby shop" with no systematic study of source signatures of average background levels and of the broad spectrum of potential approaches. Also it has not been clear that the approaches being followed would ever be practical in producing operational equipment.

In many cases though the applications for trace gas detection are quite different and orthogonal to each other, many common problems arise, e.g. (1) how does one sample a gas containing small traces of a given material? (2) how does one transmit these samples through the instrumentation without loss of the trace gases?

It seems to the panel that one laboratory with expertise in trace gas analysis should undertake all 6.1 effort to determine how to solve those problems which are common to all applications of chemical detection. The panel also felt that the mission oriented applications in the 6.2 to 6.4 categories should be undertaken by the mission oriented laboratories since the approaches to be followed would vary drastically with the problem to be solved. It also seemed to the panel that with the possible exception of mine detection and chemical agent detection there was a lack of formal requirements in the trace gas area. Obviously before further effort be expended in trace gas detection, Army requirements should be reviewed and properly defined.

The panel hopes that the conclusions and recommendations of Section III will furnish more maturity and organization to the area of trace gas detection. There is no doubt that trace gas detection needs a better program development and that uses of trace gas detection must be put in perspective with other detection techniques. Now that the lore and usefulness of the CN detector can be better scrutinized, the whole trace gas detection program should be approached more systematically and maturely.

III. Conclusions and Recommendations:

Based on our exposure to the problem, the group has arrived at the following conclusions and recommendations:

a. Conclusion: Trace gas analysis programs falls within the broad area of detection technique research.

Recommendation: Trace gas analysis detection research should be considered an integral part of the Army Research Programs on various detection techniques. The level of 6.1 and 6.2 efforts in each area should be based upon the relative merits of trace gas techniques as compared with other detection techniques. Any 6.3 and 6.4 effort should be the result of a valid mission requirement for which the trace gas techniques provide the optimum solution. The appropriate levels of effort should be determined by the laboratories in charge of each mission.

b. Conclusion: The management and direction of the development efforts in 6.2-6.4 should be similar to that employed for other detection techniques. The panel has a definite concern in this area since it is not clear that the systems discipline customarily employed by the AMC

laboratories is being adhered to in all DA sponsored Trace Gas Detection programs.

Recommendation: The exploitation of a specific trace gas detection technique in a 6.3 and 6.4 type program should be done with the aid of a thorough systems analysis that is guided by the mission constraints. Major constraints are: (1) fitness of purpose; (2) availability; (3) reliability; (4) maintainability; (5) cost.

c. Conclusion: Work in this area is fragmented among three laboratories (Edgewood Arsenal, MERDC, Land Warfare Labs).

Recommendation: The R&D work aimed at satisfying a specific mission requirement necessarily encompasses 6.1 and 6.2 efforts which are specifically related to the performance of the system in question. Physico-chemical properties of the agents to be detected, characteristics of the detection environment, factors relating to vapor availability and distribution, and the dynamics of sample acquisition are vastly different in each requirement application. Consequently, all R&D phases will have to remain the responsibility of the lead laboratory charged with the mission. However, coordination of the various efforts must be strengthened to assure the most effective utilization of data and technologies. This coordination should be accomplished at the DA staff level. Coordination at this level would assist in identifying the available resources within and outside the Government to carry out specific support tasks in the 6.1 and 6.2 categories.

d. Conclusion: Virtually all existing programs in this area are based on the current state-of-the-art technology. The achievement of

higher detectivity levels will require a breakthrough. With the possible exception of the enzyme work at Edgewood, there is no significant program aimed at a breakthrough in detecting a concentration level below 1.0 ppm. Remote gas detection by IR-optical means a program that is still in its early stages promises considerable growth potential. With exception of the old LOPAIR system and RAMAN scattering system, significant efforts are not underway in this research field within the Army.

Recommendation: In addition to the enzyme work at Edgewood, new 6.1 and 6.2 programs should be undertaken with the objective of developing fresh approaches. At the present, though we can routinely "count" individual nuclear, electronic, or photon events, there are no means of counting individual molecular events. Such an approach might be one that is concerned with surface-gas phenomena. An example of a useful extension of technology in the remote sensing area could be investigations concerning the use of tunable and nontunable lasers.

e. Conclusion: The first area in which this trace gas detection technology has been applied is in the chemical warfare/biological research (CW/BR) programs.

Recommendation: Edgewood Arsenal should continue to be the center of mission oriented efforts in the chemical warfare/biological research effort.

f. Conclusion: Two of the novel trace gas detection programs involve enzyme research and the use of bioluminescence. Enzyme work is especially promising because of its high selectivity. The panel is intrigued with the bioluminescence effort but has reservations about the validity of the observations and results that have been reported.

Recommendation: Work in both of these areas should be followed to logical conclusions.

g. Conclusion: Work on detection of drugs and explosives is in a very early stage. Very little quantitative data on the composition and vapor pressure of effluents from drugs and explosives exists.

Recommendation: Further work concerned with the detection of heroin and TNT should include a more analytical approach with controlled experiments until it is known what is being measured.

h. Conclusion: The Army Research program is generally lacking in basic analysis. This should be part of any 6.1 program. The program for the most part is applied in nature.

Recommendation: Emphasis should be shifted to more basic research. Applied research will be justified to a greater extent when requirements are more distinct and the technology is more at hand.

i. Conclusion: Even with the limited exposure that the Panel had to typical programs in the CW area, physical methods of analysis seem to have more promise of success than chemical methods. Edgewood Arsenal seems to favor and emphasize the chemical approach. (Only chemical detection methods have been developed and most current efforts are directed at small modifications of the existing techniques).

Recommendation: More priority should be placed on physical methods.

j. Conclusion: With the exception of CW/BR and mine detection programs, there are presently no formally expressed long term requirements that justify 6.2 development efforts in trace gas

detection technology. The Combat Developments Command is relatively dormant in this area. With the emphasis on air pollution control, this is hard to understand.

Recommendation: The Army must determine what its needs are and then establish requirements to guide developmental efforts. Requirements in such areas as air pollution/monitoring and control, protection of public figures, civil disturbances control, and explosive detection are urgently needed if meaningful work is to continue.

IV. Observations Leading to Conclusions:

Most of the requirements leading to trace gas techniques have come in the form of ENSURE requests. Materiel Needs Documents have supported only the chemical agent and mine detection programs. As a consequence, many of the techniques sponsored or supported by such Army laboratories as Land Warfare Labs have not been subjected to the normal management processes. From the presentations given to this group we conclude that as of this time no clear cut requirements have been defined for the military use of trace gas detection. Though we feel that the need does exist and applications will be found in explosive detection, concealed weapon detection, narcotics detection, personnel detection, etc., the problems have not been carefully studied or defined. For example, does the Army have an interest in detecting narcotics? If so, what are the minimum requirements necessary to make their detection useful? Must the detector be able to detect trace gases from a thousand foot altitude in a helicopter or by sniffing a suitcase or a sealed package? For what time duration can the instrument sample in a practical system? What is a typical scenario for the use of this instrument? Until questions such as these have been answered and a working parameter envelope has been

clearly defined for an operational and useful system, it is impossible to critically evaluate the approaches presently under study. We have found that in all cases except chemical agent detection the laboratory investigators have guessed what the Army would need without ever having received formal specific requirements.

In some respects the detection of trace gases is analogous to an electronic detection system. In the latter situation one normally considers an RF source which is emitting radiation at a given frequency and intensity. One then worries about what will be necessary to detect the signal from this source in the presence of a background, comparable signals from other similar sources and of noise which may be produced inside or outside of the detecting system. By a careful analysis of this type one can determine the bandwidth and the monitoring time which will be necessary to detect the target source.

The problem of the detection of trace gases emanating from a source is in many respects similar. The source will be emanating vapors at a given rate into the atmosphere. These vapors will be dispersed or carried in a narrow plume. The dispersal rate will depend on the meteorology. The effluents from the source will be mixed with trace gases of a similar nature (the background) and with other interfering trace gases which may give similar signals in the detecting instrument (the noise). If one knows the rate at which the source emits its signature and the nature of the signature, as well as the corresponding characteristics of competing background and noise, it should be possible to determine how long and at what rate one must sample in order to get a signal with a given signal to noise ratio. If the nature of the noise is

known, it should also be possible to determine the necessary selectivity (bandwidth) necessary to get high levels of confidence.

Hence, for any given application there are certain critical parameters which must be specified. For trace gas detection these include:

Detector sensitivity. This is the minimum amount of trace gas necessary to produce a signal with a given signal to noise ratio in the detector itself. Usually the time constants and sweep rates must be specified.

Selectivity. This is analogous to the bandwidth in an RF detector, and determines what other interfering species will also give positive signals in the detector. However, in many cases it is difficult to quantitatively specify this particular parameter.

Sampling time. This includes the rate of flow of gas through the sampling orifice.

System sensitivity. This quantity will be determined by the rate of sampling, sampling time and the detector sensitivity.

The Instrument Configuration. The final configuration of an instrument for detecting trace gases for a particular application will be very dependent on the required characteristics. If by none of the known approaches can one arrive at the required sensitivities and selectivities for the needed sampling rates and durations, it is a waste of time and money to proceed any further. If it appears that it is feasible to use chemical trace detection for the application, then the final configuration will be dependent on the parameters which we have just determined.

The usual instrument will consist of various parts some of which may or may not be necessary.

Sampling nozzle. Some means must be available to sample the atmosphere and to transmit the vapors into the detector. Up to the present time very little care has been given to either the method of sampling trace gases or of transmitting very small quantities of gases through the apparatus.

Preconcentrator. If the detector sensitivity is not sufficient for the application, it may be possible to preconcentrate the trace gas by accumulating the vapors for a predetermined length of time. In this way the sensitivity of the overall system can be enhanced.

For example, the adsorption of trace gases on clean goldfoil has been successful in the Hydronautics instrument and in the NO gas discharge detector. The surface of the gold is cleaned by heating to 400°C. Then the atmospheric sample is passed over the clean surface where the organic impurities are adsorbed on the gold surface. After the sampling time has elapsed the adsorbed trace gases are re-evolved in a short pulse by flash heating the gold surface to 400°C.

Detector. The trace gas which has been preconcentrated and possibly modified is now transported to the detector where it is analyzed. The detector could be any physical or chemical means of determining the concentrated gas.

Data processing. The data coming from the detector can be further processed to increase the signal to noise ratio and increase the selectivity.

Other factors in the final configuration of the equipment, such as reliability, maintainability, and transportability in the field must be considered.

Once the requirements have been clearly defined, we recommend that a systems analysis be conducted to determine the most promising approaches to each problem. Since trace gas analysis will probably never be a high priority requirement of the Army, funding will be limited. In order that the funding be most efficiently used, it is necessary that the most promising approaches be singled out. A systems analysis is of great aid in such a case.

Trace gas analysis is amenable to the systems analysis approaches devised for radar detection: one must consider signal strength, source, target and clutter in background as in radar applications. We believe that such a study will immediately eliminate many useless approaches and focus on others which are more promising. Other factors which should be also a part of the final systems analysis and properly weighed are reliability and maintainability of equipment. It is the general consensus of the ADHOC group that such a study will most likely lead to the following conclusions:

a. Not enough importance has been given to selectivity. If a technique is sensitive but doesn't distinguish between various trace gases, it will prove of limited utility.

b. In many cases it will be more convenient to detect a commonly occurring volatile impurity rather than the material itself. For example, in TNT the vapor pressure of the DNT is roughly equal to if not higher

than that of the TNT, and DNT is always present in commercial unrefined TNT as an impurity.

c. Often some means of preconcentration and extended sampling may be necessary for very high sensitivities.

d. Technical approaches which use physical means of detection such as spectroscopy, magnetic resonance, mass spectroscopy will often be more readily maintainable and reliable than standard chemical means such as those presently used in the M8.

Many of the basic problems associated with trace gas detection are common to all approaches. Thus in all techniques being attempted, a most troublesome step seems to be the transport of trace quantities of gases through the instrument to the detector. We recommend that the surface properties of the trace gases in question and the adsorption of these dilute gases on surfaces be more fully studied to optimize the rate of transport of these gases during their passage to the detector. Little attention has been paid to the techniques of air sampling and the proper construction and mechanical design of nozzles. For example, in heated systems the nozzle tip is often the coldest surface in the system. Hence, in many cases the trace gases to be detected adsorb on the nozzle tip and never proceed to the detector.

Other types of detectors. We recommend that enzymes systems be investigated as potential detectors. Enzymes offer promise for detecting nerve gases and are being successfully used in the FRAT system for the detection of morphine. Enzymes are specific to one type of molecule (the keyhole theory). If proper enzymes specific for a particular trace gas

could be found, such a system might provide the ultimate in both sensitivity and selectivity for sniffing applications.

Perhaps surface effects could be directly used for the detection of gaseous materials. Since certain compounds such as TNT adsorb on surfaces, it is possible that a detection method might involve concentration by adsorption on a surface and detection while on the surface itself, rather than by detecting them in the gas phase. Zeolites and claphthrates might very well function as trapping media for selective molecules.

The Study of the olfactory process. Since the dog has been so successful in the detection of mines and booby traps and since it is likely that the dog uses extensively the sense of smell, basic studies should be done on the olfactory sense and the mechanisms used by animals for detection. We recommend support be given in the 6.1 category for such studies.

For remote sensing resonant fluorescence and resonance adsorption seem to be promising approaches and we recommend that the scattering and adsorption cross-sections of potential trace gases be determined.

Standardization. We recommend that attention be given to the standardization of sensitivities and selectivities of the various detectors in development. For example, on one of our field trips to observe the bioluminescent detector in operation, members of this panel determined that while the contractor thought he was sensing heroin vapors he actually was sensing acetic acid, a common impurity in heroin. Hence, false absolute sensitivities have been claimed for this detector in this particular application. In another example, the Hydronautics explosive detector seems to detect TNT particles rather than true vapor. Because of the possible presence of impurities, it is difficult to prepare

standards at very low concentrations. Hence, often these quoted sensitivities are meaningless numbers at this time.

Though it is easy with present day laboratory technology to detect and identify individual nuclear events, i.e., individual electrons and photons, it has not been possible to extend this technology to molecules. In fact, it is difficult to detect 10^{12} molecules of any given species. Before this disparity in our abilities is overcome we believe that some breakthrough is necessary. While electrons and photons are detected in multiplier tubes which amplify by creating avalanches, it does not seem likely that these techniques are directly applicable to molecules. Some other technique of chemical amplification seems necessary. Catalysis offers such a possibility. For example, enzymes can greatly increase the rate of certain chemical reactions by acting as catalysts. Besides, enzymes are known to be highly specific to given molecules in their effect. For example, they can distinguish between stereoisomers. Enzymes might be the basis on which molecular multipliers could be developed for analysis and detection techniques.

An early application of chemical detection was in the CW/BR area and Edgewood Arsenal has had much experience in this field. However, the level of sensitivity necessary to detect a lethal chemical cloud is much lower than that needed in trace gas detection. Most lethal clouds contain agents at concentrations higher than one part per million. To detect concentrations at a part per billion is a much more difficult procedure. Although Edgewood Arsenal has done a noteworthy and competent job of detecting in the CW/BR area, these procedures are in many respects quite different from those necessary in trace gas detection.

Although enzymatic methods show promise, the bioluminescence detector described to us does not seem to show the sensitivity or specificity necessary. Also we severely question the validity of the observations which have been reported. We believe that all of the work done on bioluminescence should be meticulously scrutinized.

The work being done in some of the Army laboratories on the detection of drugs is lacking in a systems analysis approach. In many cases the chemical signatures of these materials are unknown. The volatilities of some of the materials are so low as to almost defy measurement. For example, the bioluminescent detector when used for detecting heroin is detecting acetic acid usually contained in elicited heroin.

At the present time some Army laboratories (LWL) have been developing chemical detection techniques where there is no Army requirement. Presently, there are efforts in air pollution, drug detection, and other areas where no formal requirements exist. Work should not be done where no Army requirement exists.

ASAP STUDY - TRACE GAS DETECTION

1. Name: ASAP Ad Hoc Group on Trace Gas Detection
2. Statement of the Problem: To evaluate the most promising approaches to techniques and uses of the detection of trace gases in military application. The probability of success of each approach will be assessed.
3. Considerations:
 - a. The Army has been investigating the feasibility of detecting the chemical properties of trace gases in concentrations of 1ppm or less. These techniques may be useful in detecting chemical warfare agents, explosives and mines, exhausts of internal combustion engines, people, etc.
 - b. Numerous techniques are being studied. For example, the use of mass spectroscopy is being investigated by LWL, electronic emission spectroscopy by MERDC, IR absorption spectroscopy by Edgewood, Remote Raman Sensing, etc. Other promising methods may not be under investigation at the present time.
 - c. Besides their use in pure military application such as low and mid intensity conflicts, these techniques may be very useful in civil applications such as squelching the growing number of civil disturbances, early detection of bombs and explosives in commercial airlines, monitoring air pollutants and smoke stack effluents, etc.
 - d. These methods might also find application in intelligence purposes.
4. Terms of Reference:
 - a. Identify promising techniques for trace gas detection.
 - b. Identify the techniques with low and high technical risks.
 - c. Identify the possible limits of sensitivity of each method.
 - d. Advise on new methods which should be explored by the Army.
 - e. Recommend if the efforts in this field can be more effeciently organized and directed.

BRIEFINGS ON CURRENT EFFORTS
IN TRACE GAS DETECTION8 July 1971

Physical Methods of Trace Gas Detection	Mr. Harvey Tannenbaum, EA Mr. Lee Appel, EA
Chemical Methods of Trace Gas Detection	Mr. Andrew Davis, EA Mr. Bernard Fromm, EA Mr. Sam Sass, EA
Deseret Test Center Approaches to Trace Gas Detection	Dr. Kenneth Brauner, DTC
Perspectives in Detection	Dr. Edward Poziomek, EA

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ARPA Efforts on Trace Gas Detection	Mr. Ravitsky, ARPA
U. S. Navy Efforts on Trace Gas Detection	Dr. F. E. Sallfeld, NRL Dr. F. W. Williams, NRL
U. S. Army Land Warfare Laboratories Work on Trace Gas Detection	Mr. Milton Cutler, et al, LWL
MERDC Efforts in Trace Gas Detection	Mr. James Wallen, MERDC Mr. Maryland Kemp, MERDC

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Overview of EA Program on Trace Gas Detection	Dr. Love, EA
Human Effluent Studies	Dr. Ellin, EA
Enzyme Alarm	Mr. Davis, EA
Laser RAMAN	Mr. Tannenbaum, EA
Personnel Detection	Mr. Appel, EA
Field Sampling Technology	Mr. Koblin, EA

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Advances in Trace Gas Detection at
Westinghouse Corp.

Trace Gas Detection - Status Review

Environmental Trace Gas Analysis

Dr. Garbuny, Westing-
house Corp.

Mr. Kemp, MERDC

Dr. Murrmann, USA Cold
Region Rsch & Eng. Div.

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