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4 January 1988 ENGLISH

16191

BEIJING SPECIALITY GAS RESEARCH INSTITUTE (BSGRI)

DP/CPR/85/005/11-01

PEOPLE'S REPUBLIC OF CHINA

Third Status Report*

Prepared for the Government of the Peoples Republic of China by the United Nations Industrial Development Organization, acting as executing agency for the United Nations Development Programme

Based on the work of Willard L. Ent, Chief Technical Advisor

Backstopping officer: M. Derrough, Chemical Industries Branch

United Nations Industrial Development Organization Vienna

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ABSTRACT

This third status report of the CTA for Beijing Specialty Gas Research Institute describes an eleven day visit to Beijing to monitor the activities as they have occured and to plan for the activities of 1988. The report contains revised Job Descriptions for experts required in 1988; listing of residual equipment to be received or purchased; a report on the Western Europe Study Tours of 1986 and descriptions of attempts to place PNIDO Fellows.

INTRODUCTION

This report describes the activities of the Chief Technical Advisor during his third visit to Beijing, China to review and monitor the activities of Project DP/CPR/85/005; Beijing Specialty Gas Research Institute (BSGRI). The basic report has four sections. These are Job Descriptions; Instrumentation and Equipment Purchases; Study Tours and Fellowships. The content of each section was reviewed with BSGRI personnel and they agreed with the CTA on how the information is presented herein.

A modest number of changes are requested in the report. These were reviewed and approved by the members of the Tripartide Review Committee (IPR). The TPR was conducted during the time of the CTA's visit.

JOB DESCRIPTIONS

As in prior reports of visits of the CTA to BSGRI, the changing specialty gas "climate" in China has necessitated several changes in the requirements for Experts. Listed below are all of the various experts which have been required from the beginning of the project; their current need or deletion, and the reasoning for changes as well as the proposed schedule for these experts required in 1988. The revised Job Descriptions for those experts required in 1988 are included as Annex A. The recommended personnel for the 1988 requirements are listed below. All of the below listed was reviewed with the members of the TPR Committee and received their concurrence.

- #11.01; Chief Technical Advisor.
 This report outlines the activities of the CTA during this most recent visit. A new Job Description is contained in Annex A.
 Recommendation: Willard L. Ent
- 2. #11.02; Expert in Fourier Transform Infrared Spectrophotometer. Equipment not purchased. Expert not needed.
- #11.03; Expert in Atomic Absorption Analysis.
 Expert visited BSGRI in the Fall of 1987. Project completed.
- 4. #11.04; Expert in Carbon Dioxide Purification. BSGRI personnel have been able to solve these purification problems without the need of an expert. "Specification" quality product is being produced by Beijing oxygen plant using BSGRI technology at present.
- #11.05; Expert in Food Ripening and Preservation Gases.
 Expert still needed. Revised Job Description included in Annex A.
 Projected for July 1988.

Recommendation: Dr. Wang, Chien Yi, PhD.

Research Horticulturist

Horticulture Crops Marketing Laboratory

Agriculture Research Center

U.S. Dept. of Agriculture Room 113, Bldg. 002 BARC - West Beltsville, MD 20705

- 6. #11.06; Expert in Nitrous Oxide Purification. Expert no longer needed. B.O.P. has purchased an N₂O plant which is in operation producing "specification" grade product.
- 7. #11.07; Expert in Sterilizing Gases. This product no longer being marketed or required in China. Expert no longer required.
- 8. #11.08; Expert in Equation of State for Gaseous Computer Program. Slight modifications to previous Job Description. New Job Description included in Annex A.

Projected for April 1988.

Recommendation: Dr. Robert D. McCarty

National Bureau of Standards

Division 774.30

25 Broadway

Boulder, CO 80303

9. #11.09; Expert in Standard and Calibration Gases. Slight modifications to previous Job Description. New Job Description included in Annex A.

Projected for May 1988.

Recommendation: Mr. William Derko

Office of Std. Psch. Matl's.

National Bureau of Standards

Room B 332, Bldg. 222 Gaithesburg, MD 20899

- 10. #11.10; Expert in Specialty Gas Safety and Toxicology. Project completed in November of 1986.
- 11. #11.11; Expert in Gas Blending and Analysis.

Slight modification to previous Job Description. New Job Description included in Annex A.

Projected for April 1988.

Recommendation: Mr. H. E. Lindenmoyer

2011-B Chestnut St.

Emmaus, PA 18044 U.S.A.

INSTRUMENTATION AND EQUIPMENT

Although all of the purchased equipment and instrumentation has not as yet been received at BSGRI, practically all of the purchases, as approved in the original project document (with revisions), have been completed. A review of the items still to be purchased was conducted during the TPR meeting and agreement was reached on proceeding with the final purchases. The major item(s) is the calibration standards. These were described in the CTA's report of 28 August 1986 as a portion of Annex B of that report. Mr. Derrough is aware of the items and pages and has agreed to initiate the necessary action to accomplish these purchases. There are a total of forty-seven items at an estimated cost of \$24,000 (U.S.). Also required are electronic grade and high purity grade Gas Regulators (items 1 and 2, Annex B of the 28 August 1986 report). These are estimated at a cost of \$3,150 (U.S.). A water purifier is also still required at an estimated cost of \$1,500 (U.S.); and several data books for the Toxicology and Safety Center at an estimated cost of \$1,000 (U.S.). Mr. Derrough also has the details on these latter three items and will initiate what is necessary to accomplish their purchase.

Items which have been purchased and not as yet received are the vapor phase chromatrography apparatus - scheduled for delivery in March of 1988; and the Fluids Pack "software" program which will probably be brought to BSGRI by Dr. McCarty when he serves as an expert in April of 1988.

The foregoing includes and completes all of the instrumentation and equipment purchases as originally (or with revisions) were authorized in the project.

STUDY TOURS

Two study tours have been accomplished since the inception of the project. The first included visits to many specialty and industrial gas manufacturers in Western Europe. This tour was conducted in September of 1986. Annex B is a copy of the Final Report of the Study Tour as prepared by BSGRI personnel. It is a very comprehensive report (75 pages) and describes the various aims of the tour as well as the activities during the tour in accomplishing the aims. This report was received by the CTA during this most recent visit to Beijing.

The second study tour was conducted in September and October of 1987 and included visits to the major specialty and industrial gas manufacturers in the United States and Japan as well as visits to several governmental and educational institutes which are germane to the project. The final report for this second study tour is not as yet completed. A positive result of the tour was the assistance in procuring certain of the experts required in 1988. The USA - Japan study tour also enabled the participants to expand their knowledge of specialty gas activities. When the report of the tour is received, it will be forwarded.

The final costs for both Study Tours closely approximated the Project Budget for these items.

WILLARD L. ENT CONSULTANT

Appendix A Page 1

P.O. BOX 212 EMMAUS, PA 18049

(215) 437-4310 (215) 437-4311

4 January 1988

Dr. John Chen, Chairman
Department of Chemical Engineering
Whitaker Laboratory, Bldg. 5
Lehigh University
Bethlehem, PA 18015

Dear Dr. Chen:

It was a pleasure talking to you by phone recently concerning our attempts to have Fellows from the Beijing Specialty Gas Research Institute attend Lehigh University for some advanced courses.

As agreed, I have outlined below the current thinking of the sponsoring agencies concerning these Fellows. The additional data presented is for further clarification.

- 1. The sponsoring agencies for the Fellows (fellowships) are The United Nations Industrial Dvelopment Organization (UNIDO); Vienna International Centre, P.O. Box 300, A-1400, Vienna, Austria and the China International Center for Economic and Technical Exchange of the Ministry of Foreign Economic Relations and Trade, No. 18 Bei San Huan Zhong Lee, Beijing, China. The contact in Vienna is Dr. M. Derrough, Chemical Industries Branch; and in Beijing Mr. Yao, Shenhong, Proramme Officer.
- 2. The placement of Fellows sponsored by UNIDO and a cooperating government is the responsibility of the Carl Duisberg Society, Inc., 425 Park Avenue, New York, N.Y. 10022. The contact there is Ms. Leslie Wright, Program Officer. All official relationships between the Fellows and Lehigh University will be through this society.
- 3. There are six Fellows who wish to attend Lehigh University. They wish to attend for two semesters (starting in the Fall of 1988 and concluding after the Spring semester of 1989). The original idea to have them be fully matriculated students in pursuit of Masters Degrees has been abandoned. They now merely wish to take all of the graduate level courses which can be reasonably taken in two semesters. The following are the six fellows with the disciplines they wish to study.
 - a. Advanced analytical chemistry and instrumental analysis techniques.

-2-

4 January 1988

Ms. Chen, Thifen, and

Ms. Fan, Jinwen

b. Advanced chemical engineering courses.

Mr. Luo, Gang, and Mr. Meng, Wenzhi

c. Advanced computer science courses.

Mr. Li, Wei, and Mr. Zhang, Ge

All of these UNIDO Fellows have at least "4 year" college degrees from Chinese universities.

4. All of the aforementioned fellows have studied English and all have taken the "Michigan" test and have passed it with scores acceptable to UNIDO for Fellows. If the TOFEL test is necessary for admission of foreign students at Lehigh, we will need to know that quickly as a lot of time is required to arrange for this test in China.

If all of the foregoing can be accomplished, the next step would be for the Carl Duisberg Society (CDS) to contact Lehigh. I'm sure that Ms. Wright with CDS would like to be made aware of any potential problems or pitfalls associated with placing these Fellows.

Please contact me if I can be of any further service.

Very truly yours,

W. L. ENT., INC.

Kus & Gudlew

Willard L. Ent Consultant

cc: CDS BSGRI

CTA's Report to UNIDO

FELLOWSHIPS

The institute and CDS has been having difficulty in placing Fellows. During the Study Tour in October 1987, Director Chen met with several of the professors and department heads at Lehigh University, and it was felt that all attempts should be made to, where possible, have the Fellows acquire Master's Degrees. It has since been learned through CDS and Lehigh that the potential for procuring Master's Degrees would be fraught with administrative and other problems and would therefore be nearly impossible. The appended letter* to Dr. Chen at Lehigh from the CTA is an attempt to clarify the situation as it is currently visualized by the sponsoring agencies.

* Please see Appendix A to Fellowships

UNITED NATIONS

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

UNIDO

PROJECT IN THE PEOPLES REPUBLIC OF CHINA

JOB DESCRIPTION

DP/CPR/85/005/11.01

POST TITLE:

Chief Technical Advisor

DURATION:

1 month

DATE(S) REQUIRED: May and October 1988 15 days each trip)

DUTY STATION:

Beijing, China and travel within the country.

PURPOSE OF PROJECT:

To establish a national specialty gas research and development centre at the Beijing Specialty Gas Research Institute (BSGRI) in order to enhance the national technical capability in manufacture, analysis, storage, transportation, safe handling and use, and applications technology related to specialty gases and with particular reference to their applications in electronics, medicine, food industry and environmental protection.

DUTIES:

To accomplish the purposes of the project. More specifically:

- 1. To review the progress and status of the project.
- 2. To implement the revisions to the project as agreed in the TPR of December 1987; particularly:
 - a. Review the activities of experts scheduled for 1988.
 - b. Assist in the preparation of the PPER for 1988.
 - c. Assist where appropriate in the placement of Fellows.

- d. Review the operation of and acceptability of the equipment which has been provided by UNIDO.
- Prepare a final report covering how all of the duties were accomplished.

OUALIFICATIONS:

High level scientist or engineer with extensive experience in the specialty gas industry including research and development work.

LANGUAGE:

English

BACKGROUND INFORMATION:

- 1. The development of the Beijing Specialty Gas Research Institute is progressing very well.
- 2. BSGRI personnel are desirous to have the CTA cortinue to work with them.

UNITED NATIONS

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

UNIDO

PROJECT IN THE PEOPLES REPUBLIC OF CHINA

JOB DESCRIPTION

DP/CPR/85/005/11.05

POST TITLE:

Expert in the Utilization of Gases and Gas Mixtures for Food Ripening and Food Preservation.

DURATION:

1 month

DATE:

July 1988

DUTY STATION:

Beijing, China

PURPOSE:

- 1. To develop techniques for the ripening and preservation of foods using various gaseous atmospheres.
- 2. Establish analytical methods for measuring these gaseous atmospheres.
- 3. Establish analytical (biological) techniques for measuring the adequacy of the preservation and ripening techniques.

DUTIES:

To accomplish the purposes of the project; more specifically:

- 1. Complete a medium range test (preservation) for two to three vegetable species.
- 2. Instruct in the techniques for producing preservation atmospheres in packages and containers.
- 3. Instruct in the techniques for utilizing ethylene and other gaseous atmospheres for ripening.
- 4. Deliver lectures or conduct symposia anent:
 - a. Food preservation and ripening atmospheres.
 - b. The economic benefits of utilizing gaseous preservation and ripening atmospheres.

5. Prepare a final report covering how all of the duties were accomplished.

OLIALIFICATIONS:

High level scientist with extensive experience in food ripening and preservation gases and atmospheres including research and development work.

LANGUAGE:

English

BACKGROUND INFORMATION:

- Food preservation and ripening gases are not used in China since the economic value of these gaseous applications has not been studied or understood.
- 2. The Beijing City Government has requested BSGRI to investigate the potential for preserving vegetables using industrial or other gases.
- 3. Overabundance and shortages of certain vegetables and other foods exists in China causing waste at certain seasons and serious food shortages at other seasons.

UNITED NATIONS

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

UNIDO

PROJECT IN THE PEOPLES REPUBLIC OF CHINA

JOB DESCRIPTION

DP/CPR/85/005/11.08

POST TITLE:

Expert in Equation of State for Gases Computer Program.

DURATION:

21 days

DATE REQUIRED:

April 1988

DUTY STATION:

Beijing, China

PURPOSE OF PROJECT:

To develop a computer software program for the Pressure-Value-Temperature (PVT) Blending of Gas Mixtures.

DUTIES:

Complete the purpose of the project, specifically:

- 1. Train BSGRI personnel in the use of the computer and the "Fluids-Pack" program.
- 2. Train BSGRI personnel in the use of a PVT gas mixture program for 3-component gas mixtures: specifically 4 Molar % CO₂, 0.7 Molar % Methane in Nitrogen; and 0.3 Molar % carbon monoxide, 20 Molar % oxygen in nitrogen.
- 3. Deliver lectures or conduct symposia on the development and use of the "Fluids-Pack" program.
- 4. Prepare a final report covering how the purpose of the project was accomplished.

QUALIFICATIONS:

High level scientist with extensive experience in gaseous or fluid equation computer programs and in operation of computers.

LANGUAGE:

English

BACKGROUND INFORMATION:

There are no "advanced" techniques for utilizing a computer and software for accomplishing the purpose of the project in China today.

UNITED NATIONS

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

UNIDO

PROJECT IN THE PEOPLES REPUBLIC OF CHINA

JOB DESCRIPTION

DP/CPR/85/005/11.09

POST TITLE:

Expert in Standard and Calibration Gases.

DURATION:

14 days.

DATE REQUIRED:

May 1988

DUTY STATION:

Beijing, China

PURPOSE OF PROJECT:

To establish a National Center for the production of primary and secondary calibration gas standards.

DUTIES:

To accomplish the purpose of the project, more specifically:

- 1. Instruct BSGRI personnel in the method of preparation for primary gas standards.
- 2. Deliver lectures or conduct seminars on the analysis techniques for pure gases; and describe these techniques as they are practiced by the United States National Bureau of Standards.
- 3. Instruct BSGRI personnel in the proper treatment for gas cylinders used for calibration standards; and the testing methods used for determining gas mixture stability.
- 4. Explain the equipment and accessories necessary to produce primary standards.
- Visit BSGRI's standards production facility and comment on the techniques used in producing these standards.

Prepare a final report covering how the purpose of the project was accomplished.

QUALIFICATIONS:

High level scientist or engineer with extensive experience in establishment of primary gas standard centers.

LANGUAGE:

English

BACKGROUND INFORMATION:

- "National" calibration standards are required in China.
- 2. The National Institute of Metrology has requested that BSGRI develop these standards.

UNITED NATIONS

UNITED NATIONS INDUSTRIAL DEVELOPMENT ORGANIZATION

UNIDO

PROJECT IN THE PEOPLES REPUBLIC OF CHINA

JOB DESCRIPTION

DP/CPR/85/005/11.11

POST TITLE:

Expert in Gas Blending and Analysis.

DURATION:

21 days

DATE REQUIRED:

April 1988

DUTY STATION:

Beijing, China

PURPOSE OF PROJECT:

To enhance the gas blending and analysis capabilities at the Beijing Specialty Gas Research Institute.

DUTIES:

To accomplish the purpose of the project; more specifically:

- 1. To instruct BSGRI personnel in the specialized gas analysis techniques for determining gas purities, impurities, and mixture analyses utilizing all of the normal analytical techniques; e.g.: gas chromatography, infrared spectrophotometry, atmoic absorption, ion mobility, etc.
- 2. Assist in the development of using a helium ionization equipped ultra-sensitive gas chromatograph for parts per billion impurity analyses.
- 3. Deliver lectures or conduct symposia in specialized gas blending and analysis systems.
- 4. Prepare a final report covering how the purpose of the project was accomplished.

OUALIFICATIONS:

High level scientist or technologist with abundant experience in specialty gas blending and analysis.

LANGUAGE:

Er.glish

BACKGROUND INFORMATION:

Beijing Specialty Gas Research Institute personnel need the assistance of an international expert in gas blending and analysis.

United Nations Development Programme Aid Project
"Beijing specialty gas research and development center"

DP/CPR/85/005

The report of the study tours of the project delegation $% \left(1\right) =\left(1\right) +\left(1\right) +\left($

BEIJING SPECIALTY GAS RESEARCH INSTITUTE

OCT, 16,1986

PROJECT DELEGATION MEMBER:

HEAD OF DELEGATION

(PROJECT MANAGER)

CHEN JIMING

MEMBER:

HUANG DONHTAO

ZHANG RONGZHENG

XUE HONGBAO

WANG CUIYING

GAO DEMING

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I. General situation

(I) Summarized account about the investigation circumstances

In order to impost the sophisticated technology from foreign countries positively, to promote the development of new type gas materials for our country, and to accelerate the modernized course of national economy, based on the chinese government proposal, the UNDP approved officially about the motion to establish "Beijing specialty gas research and development center" in Jan. 31, 1986. UNDP decided to offer 3 years free technical aid financially for our Institute, based on the requirements of the project activities, under the arrangements of UNIDO, the Beijing specialty gas investigation group which composes of 6 experts of our Institute went to Prance, Allied germany and England during Sept. 7, 1986 to Sept. 30, 1986 to make a technical investigation about their specialty gases.

During investigation in France (Sept. 8-14), first of all, we visited the project officers of the national trade and technical cooperation bureau of France, and followed their arrangements, the delegation group visited Air liquid Co. of France, among which we investigated the head office of that Co. in Paris and then the Clrande-Delorme high technology research center which is located near the Paris suburb, the head office Althagaz(including the specialty gas dept.), the synthetic specialty gas production plant which is located in Mitry-Mory and the electronic gas production plant witch is located in Chalon.

In the Clande-Delorme research center, we visited the modernized analytical research section, process research section and applied technology research section

in which we have seen some experimental facilities and installations including a well prepared experimental facilities of purification for AsH, which is under development stage (including environmental detection and tail gas treatment). the experiment for evaluating the efficiency of dust particles counter, the safety measurement cabinet of chemical gases, the control cabinet of quality flow, high sensitivity hydride detector and so many kinds experimental installations which are under research and manufacture. In the Mitry-Mony specialty gas plant, we visited different kinds sphisticated installations for purification of gases, automatic blending system for mixed gases, cylinder treatment devices with distinguishing features, severe quality control system. The production management and sales network which is controlled by the electronic brain and different kinds of safety transporting equipment, that plant can treat 100,000 cylinders per year, produce 250 species of pure gases and gas mixtures, the total specification is 650. it can also make up different kind gas mixtures of 12-23 components.

In the Chalon production plant which produces electronic gases specially, we investigated the pure silane production device which has a production capacity about 10T/year industrially, and the manufacture installation of gas mixtures in electronic grade . the design of such is very impact and available with high seriation.

During the period of investigation, we have held several technical symposiums with 9 experts belonging to different dept. of that
Co. to make real and deep discussions about the characteristics and
technical situations of electronic, food preservation, medical diagnostic, environmental specialty gases. we exchange ideas frankly
and they greatly inspired us.

The France air liquid Co. has more attentio about the development of specialty gases in China. they express their dense interests to go on the technical and trade cooperation. we understand the France standing point. In the symosiu, we introduced to the French colleagues about the aid background of UN., the relative policy of our government, the achivements and output, purposees of this project. Both sides have hopes for the cooperatio in the field of technology and trade between China & France. at the end of investigation, both sides have signed an intention document about eh technical exchange and trade cooperation in the field of electronic gas mixtures.

As our delegation in Allied Germany(Sept. 15-22), based on the arrangements of Duisburg association, we visited the specialty gases dept. of Messer Grieshein GMBH (abbreviated as MG Co.) & Linde Co.

Formerly, MG Co. hasn't any trade communication with China, we are the 1st China specialty gases investigation group visiting to their Co. therefore, they pay more attention to our investigation, Their chief manager of industrial gas dept. received us personnaly, and accompanied us from the begining to end. they had despatched 12 technical experts to introduce for us completely about different technical topics, such as: production management, sales activities, rare gases extraction, purification of pure gases, application of food gases, medical diagnostic gases, preparation of gas mixtures, analysis and detection safety technology, waste gas treatment, computer software, new products development. they led us to investigate different departments of this Co. such as: extraction installations for rare gases, liquid He share packageing factory, gas mixtures, quality control system, applied technical research center and new product deve-

lopment and research center etc. in the period of investigation, we, on the way, visited the air separation unit pipe net work which has the capacity of 160,000 M³ /hr and it is located in Ruhr iron industrial zone. we made deep understanding about the air separation unit which is fully automatic with a capacity 100,000 M³/hr. the gas industry of Allied Germany develops to ultra large scale and highly automatic level with a high speed, it attracts our attention.

During the period of investigation, in the MC Co., we are invited by the host to give a report to those managers and technical persons, the topic is " The development situation of China industrial gases and specialty gases ", the speech introduced more in detail about the system, distribution, production technical situation and development forecasting of China gas industry. we expressed our views about the China market demend, the possible cooperation field of specialty gasses business between China and Germany. The same occupation person of western Germany pay more attention to the development of gas industry of our country, they expressed to keep frequently about technology & information exchange with us, they liked to develop the trade cooperation with China under possible conditions, both sides determined that we will make a braft about an intention document in dealing with this discussion as soon as we meet in China, this will be a fundamental document for developing the technical cooperation after signing by both sides.

When we were in West Germany, we investigated the specialty gases dept. of Linde Co. which is located in the north of Munich. Linde Co. is a very famous air separation equipment product Co. in the world, The Linde Co. sales the air products which used air as the raw material, on this foundation, they developed different kind specialty gases products in which the rare gases

be the major part. Recently, they began the research work and manufacture of electronic gases, we mainly investigated the manufacture, detection packageing and management sales system of standard gases production and we give the invitation to those experts who are indulging in blending and analyzing the standard gases to make technical exchange with us, they expressed in suitable time, they will like to visit China as a scholar capacity.

As the delegation group in England (Sept. 22-28), based on the arrangements of British council of Britain, we visited the Disstilier Co, Co., the Gas and Equipment Ltd. and Air products Co.

The Distiller CO2 Ltd. is a CO2 special production plant of England. During the period of several ten years before, not only they developed a whole technology series about the manufacture, purification, storage, detection and complete set equipments, but also they obtained fruitful results through independent research work in the application field of CO, . The distiller CO, Ltd. has a peculiar management method, it promoted itself to survive among the keen competition between those large Companies and broaden its market, it obtained large economic benefit. During visiting, we had talked with more than 10 experts about the development and application of CO, in welding, forging, greenhouse, tobacco, beverage, food preserving, frozen, low temperature extraction etc. we obtained important advices. recently, CO, Ltd. is urgent to find new trade partner, therefore, they expressed warm welcome to our investigation. we understood their attitude, in the symposium, we suggested the 1st step of technology and trade cooperation is to invite one expert of purification and application of CO, in the UN expert capacity to go to China making the technology exchange.

President of Distiller CO,Ltd. agreed our suggestion.

The Gas and Equipment Ltd. located in the north sea margin city, Aberdeem of England. it is the largest specialized He gas filling factory. in Europe. we, mainly, investigated the share packageing system, the He gas transporting, the analyzing, the detection and application. because the boiling point of He is 4.2K, therefore the storage and packageing is a key technology in gas industry, At present, our country can't produce large scale He storage and packageing equipments industrially, He supplied to users by means largely of gas state transportation. this is non-reasonable in economic view. Gas and equipment Ltd. hopes to sale liquid He to China through their branch CO. in Singapore, we expressed our intention to consider their opinion as soon as we be back to China.

The England air products CO. (abbreviated as AP CO.) is a branch of America air products chemical institute (abbreviated as APCI) in England, we visited mainly the specialty gas plant of that CO. located in Crew, the purification installation of tail gas treatment device, blending system of gas mixtures, safety devices and analytical detection instruments etc. the factory can produce 120 kinds of pure gases, more than thousand species of gas mixtures. the products sold to the whole England. At present, this plant is now undertaking the technical reforming, we were introduced that the investment used for technical reforming was equivalent to the sum investment of foregoing ten years, by means of forecasting, in next year, the investment will be 4 times of this year, it is planned to be fully automatic in 1988, each worker may be linked with the computer terminal and chief data base, by means of forecasting, after this project is finished, the production rate

of the whole plant can increase 5 to 10 times, this CO. understands that if you want to keep the predominant attitude in the keen competition, you must develop new technique constantly and keep the high working efficiency.

We had stayed more than 20 days in western Europe, we visited 6
gas companies of England, France, West Germany. through
investigation, symposium and technical exchange, we obtained a general
understanding about the management system, production scale and
technical characteristics of western Europe gas industry. in the mean
time, we obtained a new outlook about the development trend of
international gas industry through analysis and comparison. we new
more clearly about the managerial & technical field difference with them,
and find the developing direction and route of our specialty gases
business, these will, it is very important, help us to complete our
project tentative idas and determine an available development schedule.

During investigation, through the contact with the staff members and technical persons of several foreign companies, we introduced the present situation of our country gas industry, to propagate our present open policy, to deepen mutual understanding, promoting mutual friendship, creating a well atomosphere for international technique and trade cooperation. in the mean time, it supplies available conditions for carrying out the UN. project smoothly and also we have done research works about the technology imported from other country, invitation of experts, training of peoples, type selection of instruments and equipments.

In short, under the direction from the UNIDO representative in China and China international economic and technologic exchange center

under the warm support and organization of UNIDO, Prance technical industrial and economic cooperation agency, Duisburg association of Allied Germany, England culture center, and through the whole delegation member endeavourness, we obtained fruitful results and achieved the desired purposes, therefore, for this investigation in western Europe, we express our scincere thanks to the above stated organization and to those ladies and gentlmen who gave us very warm services and we gave kind regards to them.

- 2. General situation of the investigated unit
- (1) Prance L'air liquide

This CO. which is the representative of Prance gas industry and low temp. engineering established in 1902, and also it is one of the world largest gas group, this CO. established 130 branches in America, west Germany, Italy, Canada, Japan etc. 55 countries. it has a total about 430 factories. total employee is 25000. this CO. has 4 functional depts,: 1) industrial gas and specialty gas dept.. 2) Chemicals and other products dept. 3) Engineering design dept. 4) Sales dept.. the 5 research centers are: 1) high technology center, located in the west suburb of Paris 2) Low temp. technique research center, located in Richomeat of Prance 3) Liquid air gasresearch center, located in Chikuha of Japan 5) gas synthetic technique research center, located in Chicago, U.S.A. The products of this CO. are industrial gases, specialty gases, different kind chemicals, fertilizers and medical diagnostic gases. besides this, it can supply gas seperation and purification equipment, blending installations of gas mixtures, analytical detection instruments, storage tanks, truck lorries, low temp. cryogenic and low temp. grinding equipments.

until now, this CO, sales 106 kinds of specialty gases, but in the major part, they bought material gases from other CO. and purify it. very little amount to be manufactured directly by its own plant. the total sales amount is 200 hundreds of millon, among which the gas products are 67%, welding products are 11%, engineering constructions are 4%, chemicals and pharmaceuticals are 18%. in regarding the sales area distribution, it is in Europe 64%, US, 23%, Africa 1%, Asia and Austrilia 12%.

(2) West Germany Messer Greshelm CMBH(abbreviated as MG)

Mg CO. established in 1935, it is a main part of Hoechst chemistry group of Allied Germany, its headquater is located in Dusseldorf, the production and development base of spacialty gases is located in Duisburg, west Germany. the CO. has employees 7585, the total sales amount is 20 hundreds of millon Mark which occupies 42.7% of the sales market of west Germany. this CO. used 6%-10% of its total profit for research and development work. in 1985, the sale amount percentage of MG CO. are: iron works 26.5%, iron processing 10% mechnical engineering 7.2%, automobiles industry 4%, marine industry 1%, chemical engineering 10.2%, electronic industry 4.7%, . the other industries 6.9%, food processing 7.2%, ARCHITECTURE INDUSTRY 2.4%, energy source 3.8%, medical treatment 6.5%, public services 1.9%, foreign trade 7.7%.

The CO. divided to 3 depts. 1) industrial gases dept. 2) welding and cutting dept. 3) sales dept., this CO. possesses 18 gases factories, 130 thousand cylinders, 150 railroad tank cars, and possesses 3 big industrial gas transpoorting pipe nets in Luhr, Kalon and saar in each 5-7 KMaverage, there is a gas supply station of that CO.

MG CO. has branches in Europe, north America, Japan. the acting agency in HongKong is sonfu CO.

MG CO. started the development activites for specialty gases in 1960, the total sales amount of specialty gases is 2 hundreds of millon in 1985. the main poroduction realms of specialty gases are:

1) using air as the raw material, to purify O₂, N₂. Ar. and other rare gases ?) Importing Ne gas from U.S.A. after purifying, share packageing to users. 3) N₂ purification 4) purification and blending of other inorganic gases 5) purification and blending of alkane gases 6) supply those specialty gas equipments, apparatus, valves and pipe fittings. the main users are: medical treatment, lightning, electronics, vehicles, chemical industry, environment portention etc. Since 80 century.

MG CO. gathered the main research work in the following 3 aspects to tackle key poroblems: 1) Develop the high quality electronic gases serviced for 1M-4M-ultra large scale integreated circuit 2) Develop the 7th grade ultra high purity gases (namely 7N) 3) Application of technology research works.

(3) Western Germany Linde CO. This CO. established in 1879.

it is a translational cooperation to produce specially about the air separtion installations and H₂ share packageing equipments, it is composed by more than 50 branch companies, the total sales amount is 36.2 hundreds of millon in 1985 (estern Germany). total employee is 19,000 (westren Germany is 14000). Western Germany Linde divided to 4 depts.: Engineering construction dept., industrial gases dept., cryogenic dept. material treatment and hydraulics dept.. The industrial gases dept. is located in the place Hollrieylskren which is 25KM from Munich, the sales amount is 5 billon Mark. The specialty gas factory is located in Lohhof of Unter-Schleibheim near 120KM from Munich. this plant can produce 120 kinds of pure gas which can be poroduced by buying raw gases to purify. the whole plant has 360 employees among which there are 80 persons to produce specialty gases, 50 persons for res-

-earch works, 30 persons for sale activites, 13 persons for analyzing and the rest is indulging in the production of industrial gases.

(4) The Distillers CO, CO.

This CO. which was merged by 6 small companies established in 1928, it engaged mainly about the production of gas cylinders. in 1930, it transformed to produce dry ice and participated to Imperial chemical industries (IGI), during 1949, it supplied the users the purified CO₂ positively, the history of producing CO₂ is about more than 40 years, the Distillers CO₂ CO. announced to be independent in 1954, it began to produce plastic products since 1960 centuries. in the midst of 70 centuries, the chemicals production part was bought by other CO. the remaining part is only the CO₂ production system. after reforming, expanding and improving, this CO. became the largest CO₂ production CO. in Europe.

At present, the company headquater is located in Redhin which is near west suburb of London. the company has employees 700, it has 4 CO₂ product companies in England. the total turnover is 130 millon pounds in 1985, the main business fields of the company are:

- (1) Gas, liquid, solid state CO₂ products in different purity.
- (2) Purification installation and the fittings in series. including valves, instruments, pipe fittings etc.
- (3) Supply the special technique and equipments to use CO₂ in medical treatment, welding, forging, plant cultivation, tobacco industry, low temp. extraction, food quick frozen, beverages etc.

Recently, this CO. sales the whole technical design system & whole set production device of CO, to Egypt, Halasiz, new Zealand.

A)

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(5) England gas & equipment Inc.

This Co. estab? shed in 1970, the main managers & technical person came from England Oxygen CO. & air products CO., its service items are share filling, storage, blending, deteection and also producing liquid He equipment and those special equipments & instruments for the application of He which was imported from U.S.A. in detection, medical treatment, weldling, super conduction, nuclear magnetic resonance, diving in deep sea. this CO. is the Largest He gas share filling plant in Europe. It occupies 90% of European market and it has branches of factory in Norway, Singapore, Bombay of India.

(6) England air products CO.

This CO. is a branch of America air poroduct & chemicals Inc. in England. the head Co. located in Arlington of Pennislavia, established in 1940. This Company has sub-companies in 13 countries of the whole world. it has 16 gas plants. total employee is 162000, fixed assets is \$19 hundreds of millon.

England air products company established in 1958, its headquater located in London. its main business fields are: 1) Industrial gases, specialty gases and medical diagonostic gases 2) gas separation, purification, liquidification, storage and transportation, poackage, recovery equipments and installations 3) Chemical products 4) Engineering design and construction, and it also produces waste water treatment system equipments, welding equipments, low temp. cryogenic equipments used in laboratories etc.

This company wivided to 5 functional departments: 1) Gas department

2) process system department 3) Financial department 4) Personnel and
labour department 5) Market department, among which, the gas department
divided also to specialty gas department and air product department.

Total employee in specialty gas department is 130, in air product department

plant and a Crew plant which is in the middle part of England. the Crew plant occupies an area about 255 acres. total employee of it is 63, among which technicians and labourers are 55, staff member 8. The Crew plant indulges mainly in the share filling, purification and blending about specialty gases, the sales amount distributed to: chemical industry 22%, engineering construction 20.8% food 6.6%, electronic 31%, others 19.6%.

Table about the sales amount of the world important gas companies in 1984

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serial No.	Name of companies	Sales amount in 1984 (millon mark)	market possessing t
1	Lair Liquide company (Prance)	4253	17
2	BOC company (England)	3705	15
3	UCO company (USA)	3619	14
4	APCI company (USA)	2793	11
5	MG company (Western Germany)	1192	5
6	AGA company (Sweden)	1135	5
7	Nippon sanso company (Japan)	969	4
8	Linde company (Western Germany)	908	4
9	The sum of other companies in the world	6191	25

II. Technical situations of specialty gas in western Europe

- (1) Purification technology about pure gases
 - 1. International concept of pure gas at present
 - (1) Classification of pure gases

Pure gas is also called as unit gases which has two kinds of classification.

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ture, it is divided to 3 species in broad sense, a) simple gas substances b) gas inorganic compounds c) gas organic compounds,

It is divided to 5 species in the narrow sense, a) organic gas b)

INORGANIC Gas c) halogenic carbon gas d) isotopic gas e) semi-conductor gas (Because this species has strong special usages, customarialy, it is deemed as one species individually).

2nd kind: classified in according to the chemical properties of gases, it is divided to 5 species, a) oxidizing gas, it can't ignite spontaneously, but it can help ignition. such as O_2 , air CL_2 , b) inert gas, it has no reaction with other substances under normal temperature and pressure. such as CO_2 , He, Ne, Ar, F, Xe, N₂. etc. c) combustible gas, the mixture of gas and air can form a combustible mixture under normal temperature and pressure when the volume ratio=13%. or when the volume ratio>12%. such compounds as $Sill_4$, PH_3 , P_2H_6 , CO, CH_4 , SiH_2CL_2 are combustible. d) corrosive gas, compounds as HCL, NH_3 , H_2S , BF_3 , NO, So_2 , PCL5 are erosive to metal structures either it exhibits with H_2O or not. e) Toxic gas: compounds which are harmful to human body as chemical hazards or causing death, such as BCL_3 , COS, C_2H_2 , HF, HI, H_2S etc.

The advantages of this kind classification are easily to distinguish
the gas characteristics and to facilitate in application. the shortcomings
are duplicate in classification because some gases possess several
characteristics.

It is reported, at present, Species of pure gas nearly 259 kinds in the whole world. Different companies in Europe can supply more than 200 kinds pure gases.

(2) Species of pure gas

Each kind pure gas divided to several species, as in France air liquid company, it induced pure gases used in semi-conductor industry to 40 fundamental species, the applications can be divided as

follows: silicon intermediary gases: SiH4, SiH2Cl2, SiCl2, SiP4.

Mingled gases: λSH_3 , PH_3 , B_2H_6 , BH_3 , PP_5 .

Etching acid washing gases: HCl, Cl₂, CP₄, C₂P₆, C₃P₈, SiP₄, NP₃, BCl₃, C₄F₈,SP₆.

Intermediary gases: Ar, He, H_2 , N_2 , O_2 .

Mixed gases: NH_3 , CO_2 , N_2O , $Ca(CH_3)_3$, $Al(CH_3)_3$, $Al(C_2H_5)_3$, $Te(CH_3)_2$, $Sb(CH_3)_3$, $In(C_2H_5)_2$, H_2S , H_2Se , GeH_4 , COS, IH, WP_6 , NO.

(3) Specification of pure gas

Each species of pure gas divided to several specification, the principles of classification are different in different companies, but it mainly obeys the following principles:

- a. Classification in according to the pure gas grade.
- b. Classification in according to the ex-factory detecting level (including the accuracy of detecting instruments and real methoding detection as: Is it a routine detection or detected one cylinder by one)
- c. Classification in according to the ingredients to be controlled
- d. Classification in according to synthesization of the above factors.

Usually, in Western Europe, they determine the pure gas quality standard in according to the purity grade, it is divided almost as the following 5 grades:

- a. Research grade: In using the most precise method to purify, generally, the package purity is greater than 99.999% and also to provide the total analysis of impurities mainly it is used in the most severe situation, like determination of partial pressure, spectrometer absorption spectrum, calibration of gas chromatography and the research and development of light fibre and super scale integrated circuit.
 - b. Super high purity grade: namely, it is used in Chromatography

to analyze the carrier gas used as diluting gas of doping gas, controlling gas atomsphere in semi-conductor industry and to prepare environmental supervision standard gas etc. generally, the purity is 99.999%.

- c. High purity grade: used for special welding, controlling gas atmosphere etc.. generally, the purity is 99.99%, THC < 5pm, H₂O < 5ppm.
- d. Zero point grade: used generally in calibration about the zero point of high sensitivity instruments and meters. generally, the purity is 99.99%, but there is severe requirements about several special impurities in pure gases as follows: THC<0.1ppm, $\rm H_2O<2ppm$.
- e. Industrial grade: it denotes geneally such pure gases used in industry, the purity is generally lower than 99.99%.

Because the electronic industry has special requirement, about the gas quality, some components classify the electronic gases into 3 quality grades based on the availability of the quality of electronic gases to the large scale integrated circuit.

- a. Electronic grade pure gases: suitable for the production of medium or small scale integrated circuit (1K-4K).
- b. Electronic grade high purity gas: suitable for the production of large scale integrated circuit (16K-64K).
- c. Electronic grade super high purity gas: suitable for the production of super large scale integrated circuit (256K-4M).

Owing to the diffeent functions of electronic gases, therefore there isn't any integrated regulations about the same grade of different electronic gases purity.

The Messergreheim company in Western Germany considered "N" as the grade, it divides pure gases to 7 grades, i.e. 1N-7N. at present, that company just undergoes the research work and manufacture of 7N super high pure gas.

Summarized account about the technology of purification of pure gases

It is reported, at present, the France air liquid company has 120 gas factories, British oxygen company has 100 gas plants, the American air products and chemicals company have (including England AP company)

166 gas pants. the Linde company of Western Germany has 29 gas factories and 17 acetylene plants, the Messergreim company of Western Germany has 18 gas plants (in native country, all of those gas plants can produce and purify about more than 200 species unit pure gas. It was, in major part, through the traditional process, extracted from air, natural gas, tail gas of petrochemical industry and remaining gas of synthetic NH₃ plant, the pure gas produced by gas company bought the material gas from relative chemicals company and purify it. It is rarely to prepare directly. the purification process of different pure gas products are as follows:

1. Distillation method

First of all, to condense the mixture to liquid and then separate it in according to the distillation temperature, all of the high purity air products can be obtained through low temparature distillation method. some companies adopt multi distillation method to obtain the high purity gases, as MG company in Western Germany, they extracted Ar, Kr, Xe from air in using the above method.

2. Freezing method

Pass the gas mixtures through cooler or reversible exchanger to degrade the high boiling point ingredients by solidification. this method is used usually in the pure pre-treatment of purification technology, by means of freezing to degrade the water, CO₂ impurities, at present, the England distillers (CO₂) company has developed

the new technology of extracting CO₂ from fumming chute by means of freezing method.

3. Partial condensation method

Based on the different condensation temperature of different components of mixed gases, the high boiling point ingredients will condense out firstly in condensation process, so that the gas will be purified. such as the refining process of CL₂ in MG company is based on this principle to do the purifying treatment.

4. Absortion method

In using a liquid absorbent which has different solubility to those components of gas mixtures, it can absorb one or more component selectively to achieve the purpoose of gas purification. at present, the England AP company uses the hot K_2^{CO} absorbent to recover CO_2 , industrially its purity reaches 99.997.

5. Adsorption method

Use the characteristics of gas accumulation on the solid surface, to make the impurities adsorbed on the surface of the solid adsorbtion agent, then it is easily to be taken out.

The adsorbtion method can be divided to low temperature adsorption and varying temperature adsorption. the low temperature adsorption is to use the temperature effect adequately in adsorption process, based on the components and quantity of impurities, select the suitable adsorbent to make adsorption under low temperature. At present, the traditional purification process for obtaining high purity H₂ in the European large companies are almost to use the low temperature adsorption. besides this, it is repoorted that the production of PH₃, H₂S, C₃F₃ etc. In air liquid company of F rance, MC company of Western Germany, AP company of England are also adopting the low temperature adsorption method.

Adsorption method under variable pressure is to use the pressue effect in adsorption process. that is to use the principle making the adsorption quantity of one substance in accompanying with the increasing of partial presure of that substance. During recent several years, the Western Eupropean countries begin to use the adsorption method under variable temperature to produce high purity H₂. the adsorbents are activated charcoal. molecular sieve, activited Al₂O₃ etc. the purity of H₂ reaches>99.999%.

Besides this, the Western European countries are also using the adsorption method to extract high purity Ar, at present, it forms a serial installations, the adsorbents used are spongy titanium, Zirconium. Aluminum 16 and artificial synthesized Zeolite which is treated specially, the Ar purity reaches 99.999%.

6. Catalytic method

This method is by means of suitable chemical reaction to transform the impurities to harmless compound which exists in gas state or to tranform it to such kind compounds which are easily to be taken out, at present, the purification of SiH_4 , Si_2H_6 , AsH_3 used this method.

7. Thin membrane permeable method

Some thin membrane which are made by high polymer has the characteristics of diffusing different gas components selectively. in order to get rid off some impurities. at present, this method is widely used in the recovery and purification of He. such as AP company adopts tetrafluro ethylene—hexa fluro propane membrane to concentrate He in natural gas. BoC company adopts central cavity fibre installation to separate He in natural gas. through 4 stages concentration, it can raise the content of He from 0.5% to 99.99%.

The Linde company used a 2.5 u thickness poly-tetrafluro-ethylene to make a diffusion installation which can treat the natural gas which contains He 0.45% effectively.

8. Based on the different properties of different gas materials, the processes with special features are synthesized from those methods stated above.

Based on our investigated intession about big gas companies, we give a summarized account shout the main features of pure gas purification technology, and its developing trend as follows:

- (1) Pay attention to collect different sources of raw materials to develops new process line positively. for instance, In Western

 Germany, the raw material sources of CO₂ are: boller waste gas, brewery waste gas, regenerated tail gas from chemical fertilizer plant, waste gas from iron works, coal gas underground, natural gas, recovery of methane gas etc. they develop more than 10 processes lines including absorption, adsorption, freezing, permeable membrane etc. those processes are reliable from economic view, suitable measures to local conditions, comprehensive utilization. at present, the high purity CO₂ reaches 6N among wheih H₂O<2-3ppm.
- (2) Seize the new purification method, complete and perfect the traditional purification technique, such as AP company developed the rapid variable pressure adsorption method (RPSA), its pressure cycling period is within 20 seconds, the production capacity of unit adsorbent is greater than routine PSA. through unit bed, it can be operated contonuously, recently, a new high efficiency prrification method laser purification method is developed, it uses a fixed wave length laser to irridate so that the impurities be excited and be taken out after reaction or dissipation, the main advantage in comparing with the

traditiona! purification technology is that can excite any component if the mixture be with high selecitivity. In abroad, at present, some companies use ultra violet light to decompose the impurities AsH3 and PH₂ of silane to obtain satisfactory effect.

- (3) Pay fully attention to explore the fundamental research work about the physical properties data of gases, such as to strengthen the experiment of gas liquid equilibrium, thermodynamic properties experiment, it summarized voluminous precise and real fundamental data, created available conditions for the process experiment and engineering design.
- (4) Common use the computer to analyze different kind data and different operational conditions in order to make the reasonable economic optimistic design through the best design parameter and operational conditions.
- (5) Deep reserch the package material used in distiller, the absorption liquid, the adsorbent agent and permeable membrane. find the excellent, cheap price new species through one batch by one batch. promote the gas purification measures more deeply.
- (6) There is a batch of specialized purification equipment plant which can supply the purification equipments, pipe fittings for special use, valves, instruments and packageing vessels from those gas companies, so that the advanced process plot can be put in practice soon.
- (7) The complete technology is perfect even those technologies including analysis, package, transportation, environment having high level development, it can satisfy the basic requirements of purification technology.

What we have stated above are the main reasons that Western Europe has a predominant attitude about purification Technology, in other words, these are why our gas industry be lag behind of quality. China gas industry should poay more attention to this and must hold a real position with suitable technology as soon as possible.

- 3. Analyze the typical purification process
- (1) SiH,

In the Chalon electronic gas plant of France air liquid company, we have seen a set of SiH₄ production installation with a capacity 10 t/year. it used SiCL₄ and LiH as the raw material to produce SiH₄ through catalytic reaction. The crude gas was purified by condensation and adsorption, and finally, filled it under liquid N₂ temperature. The reaction equation is $4LiH + SiCL_{\frac{400-410 \text{ c}}{4 \text{ acid solution}4}} + 4LiCL$ the final product has two specifications:

i) epitaxy grade

$$0_2 \leq 5ppm$$

ii) Chloride grade

$$0_2 \leq 5ppm$$

the crude SiH_4 contains 2% Si_2H_6 .

In regardign to this installation, its process is reasonable, construction is compact, operation is convenient. automatic level is rather hight. the building adopts light construction materials, frame construction. Indoor uses negative pressure treatment with high sensitivity safety alarm system.

(2) AsH₃

In the Cloude-Delorme research center of France air liquid Company, we investigated a set of AsH₃ purification installation which used As₂Zn₃ and H₂SO₄ as the raw material, by means of chemical reaction, the 3N crude AsH₃ was obtained, and then, in the multistage low temperature adsorber which used molecular sieve as the packing to take out different kind impurities, finally, it was condensed and filled under liquid N₂ temperature, the reaction equiation is:

$$As_2Zn_3 + 3H_2SO_4 - 2AsH_3 + 3ZnSO_4$$

This installation adopts 10L glass bottle as the vessel, use the adding quantity of ${\rm H_2SO_4}$ to control the reaction speed. the reactor must be put under negative pressure, the laminar flow protection is used. In the room, the environmental detection alarm instrument with several contact points are installed. it is reported, this method can produce AsH₃ with a purity 5.5N. The impurities content are: ${\rm H_2O} \leqslant {\rm lppm}, \ {\rm D_2} \leqslant {\rm lppm}, \ {\rm CO} \leqslant {\rm lppm}, \ {\rm CO}_2 \leqslant {\rm lppm}, \ {\rm CH_4} \leqslant {\rm O.5ppm}, \ {\rm THC} \leqslant {\rm 3ppm}.$

At present, the france air liquid company just makes this pilot plant installation to be magnified for design. it is estimated in the next year to form production capacity industrially. at that time, this will be the 1st pure AsH₃ industrialized production installation.

(3) HCL

In the chalon electronic gas professional production plant of Prance, we have seen a set pictures and flow sheet of producing NCL

which was operated in 20 years ago, that process used the freezing method to eliminate the free H₂O and then used the molecular sieve to adsord. finally, used the distillation method to take off the light components. However, this installation was abondoned, but it showed the route that HCl purification process of France gas industry had passed, and also it embodied their development pulse of technology thought. therefore, it gives us more pratical significance about our research work. at present, although the Prance air liquid company can supply to users the 4.5N electronic grade of high purity HCL, but they are still to find new sources of material gases in order to decrease the unit cost, increase the economic profit. In the Claude Porms research center, we saw a set installation for purification of HCL. this installation adopted multi-low temperature adsorption method, used the high efficiency, anti-corrosive, artificial synthetic molecular sieve to take off those impurities in HCL, such as: H₂O, 0_2 , H_2 , THC etc. the process is simple, the construction is compact. it is reported, if this method went success, it could obtain 4.5-5N high purity HCL, among which water content < 10ppm. (at present, the normal HCL product contains $H_2o < 20ppm$), but they didn't introduce for us what were the components of HCL raw material gas and the construction of molecular sieve.

(4) SP

SF₆ 99,9% 02+N2<400ppmv, CF4<400ppmv, HF<1ppmv.

(II) Manufacturing technology of gas mixtures

1. International concept of gas mixtures

The gas state mixture which is mixed by pure gas is so called gas mixture, the proportional range of gas mixture is from ppm to 50%, each kind gas mixture is composed one kine of background gas (hereinafter called as main component) and one kind or more than one kind doping gases (hereinafter called as sub-component), now, the concept of gas mixture in western European countries is stated briefly as follows:

1) Clkassification of gas mixtures

Usually, the international classification of gas mixtures is in compliance with the usage, it is classified to 3 kinds:

a. Ordinary gas mixture

It denotes, in general sense, those gas mixture which can be used its physical, chemical and biological properties, such as, gas mixtures which used in electric light source, food profession are belonging to this kind.

b. Material gas mixtures

It denotes, in a general sense, those compounds which can be used as the chemical reaction material of gas mixtures. such as, those gas mixtures used in the manufacture of semi-conductor.

c. Standard gas mixtures

It denotes, in a general sense, those components which can be gas mixture used as the primary reference substance, such as: gas mixtures used for calibration of instruments and apparatus.

In the narrow sense, gas mixtures can be classified into several kinds in according to the concrete usages, such as, waste gas mixture expelled from motor cars, environmental, detection gas mixture., gas mi-

xture used for calibration of instruments, food preserving gas mixture, HC gas mixture, laser gas mixture, leakage detecting gas mixture, electronic industry gas mixture, light source gas mixrure, medical gas mixture, water detection and monitoring gas mixture, radio—active and stable isotopic gas mixture, special carrier gas mixture, sterilize gas mixture, welding gas mixture, high pressure gas mixture and other gas mixtures, such as, catalytic gas mixture, melting oven atomospheric gas mixture etc.

Each large kind gas mixture can be divided to several small special kinds, such as, the medical gas mixture can be divided to gas mixture for calibration the blood gas analytical instrument, lungs diffusion gas mixture, surgical laser gas mixture, biological atomospheric mixture, tissue equivalent gas mixture, diving gas mixture, radioactive isotope gas mixture etc.

(2) species of gas mixtures

Each special kind gas mixture divided to several species in accordance with the different components, such as: take the medical gas as example: (i) class, medical gas mixture (ii) special class: calibration gas mixture for blood gas analyzer (iii) species CO_2/N_2 and $CO_2/O_2/N_2$.

(3) Specifications of gas mixtures

Each kind gas mixturecan be divided to several specifications, the principles to classify the specifications of gas mixture in different countries are as follows: i) proportion of components ii) purity of material gas iii) blending method iv) analytical precision v) summary conditions about the above factors. In regarding to this problem, the standard in different companies are not the same.

It is reported by the chief manager of thespecialty gas company of Prance air liquid company, the Mitry-Mory plant, at present, can

compound 300 kinds fo gas mixture. the total specification is 3000. total species is 1500.

(4) Quality standard of gas mixture

The 3 important targets to evaluate the quality of gas mixture are analytical precision, material gas purity and blending tolerance. based on the above stated targets, gas companies like to classify the gas mixtures to several quality grades, for instance, the France air liquid company classified the gas mixture quality to 5 grades.

- a. Standard grade: using the gravimetric methos and 4N-5N components to prepare the precision obtained by balance weighing, do not
 make the analytical calibration, it is considered as standard value
 to transfer directly.
- b. Super precision grade: using the dynamic method to blend, compound it with 4N-5N components. the calibration of the deviation between ideal gas and real gas uses the electronic computer, after blending, use the precise instrument to do the analytical calibration.
- c. Precise high purity grade: using the partial pressure method to blend, such as the 4.5N component, pasblendingand controlled by computer after blending, use the common instrument for analytical demarcation.
- d. Precision grade: using the partial pressure method to blend, such as the 3.5N component gas blending and controlled by computer after blending, use the common instrument for analytical demarcation.
- e. calibration grade: using the partial pressure method to blend such as 3.5N component gas blending, sampling to do the analytical demarcation.

Quality standard of gas mixtures in Air liquid company of France

Quality grade	analytical precision (relative)	component gas purity	<pre>blending tolerance (relative)</pre>
standard grade	± 0.1 - 1%	≥ 99.995%	<u>+</u> 10.
Super precision grade	+ 18	≥ 99.995%	+ 1%
Precise high purity grade	± 2 - 5%	≫99.995%	+ 5 - 10%
Precision grade	± 2 - 5%	≥ 99.951	<u>+</u> 5 - 10%
Calibration grade	+ 2 - 5% (sampling analysis)	> 99.95€	+ 5 - 10%

2. Summarized account about the blending technology of gas mixtures

The key points of blending technology, at present, are: 1) research

about mixable property ii) the computer soft ware development of revising ideal gas iii) research of blending method.

(1) Research about mixable property

Whether it is possible to implement the predicted blending plan smoothly or not, first of all, it is decided that all the gas components to be blended can be mixable or not. under the following circumstances, those gas components can not be blended.

a. Gas components have chemical reactions during blending,

such as: NH₃ can react with HCL, HBr, F₂, CL₂, Br, CO, BCL₃.

 NO_2 can react with $\mathrm{N}_2\mathrm{O}$, organic compounds, P_2 , CO_2 , Br .

CO₂ can react with NH₃.

therefore, the gases stated above cannot mingled one another.

b. Gas components have reactions with gas cylinder, such as: CO can react with Cr, Ni, Indium steel to produce carbonyl nickel which has the explosive hazard.

 CO_2 , P_2 , HBr, H_2 , HCL CH_4 , dichlormethane cz. + ct with aluminum.

Those gases stated above can't match with relations a cylinders.

c. In the gas components for blending, the liquified gases are

in the range of condensation, such as: propane (C_3H_8) , Propylene (C_3H_6) , Butane (C_4H_{10}) , ethane (C_6H_{14}) etc. the critical temperature of those gases are greater than 10° C, it belongs to low pressure, liquified gases, there are limitations about the filling pressure as soon as it is in blending.

d. The blending proportion of oxidizing gas and combustible gas are in the combustible range.

under above conditions, there will happen the dangerous of combustion and explosion.

In accompaning with the development of different new technology, there are more requirements to the kinds of gas mixture, hence, it promotes the research work about mixable characteristics to be more important. Since 60 centuries, Gas companies in western Europe put more human power to do the systematic research works. Such as: France air liquid company, Western Germany MG company, England BOC and AP companies. they edited their gas blending handbook and sa ty handbook, and put it into electronic brain for suing intermittently to guarantee the safety of blending process of gas mixtures. at present our country is lack of such systematic research.

(2) The computer software development about the deviation between ideal gas and real gas

The most widely used blending method is still the partial pressure method in the owrld. in order to guarantee the precision of blending gas, it must correct the state parqueter of ideal gas, so that the max. limit will approach the real gas. Since Van Der Waals's equation was published in 1873, a long large amount data about research of real gas have accumulated and obtained important progress. Different gas books published in the world. But all of those calculating methods are tedious prolixity and not convenient to use.

In according to the introduction by England AP CO., before 70 centuries, their blending gas shop had 2 or 3 persons to correct the date of ideal gas specifically, not only the work was laborious, the efficiency was low, but also so many mistakes had made. in order to modify the backward condition, to live in the keen competition circumstance, the big gas CO.in Western Europe integrated large amount man power, materials, no regarding the cost to storm the technology fortification. before 70 centuries. Those gas CO. utilized the different kind informations of gas physical properties public issued internationally to integrated the self-experimental data, through unremitting efforts, they developed the soft ware about correctness of the deviation between ideal gas and real gas independently. in this case, it raised the precision and effectiveness of blending gas in a large step. in othe rwords, it produced a revolution about blending technique. It is reported, the time in completing this modification was as follows: France air liquid CO. 1972, England AP CO. 1975 (Use the America soft ware head CO.), Western Germany MG CO. 1976. the computer soft ware is still the know-how of those companies. they keep secret one another and do not make any technical transfer. at present, in our country, we still haven't the method using computer ot blend. it is estimated we are lag behind about 10-15 years in regarding this import ant technology in comparing with those Western advanced countries.

As we investigated the CREW spacialty gas plant of England Ap CO., the technician operated the computer to allocate the command on site for us, in order to keep secret, all the computer soft ware of that CO. stored in London headquater. when the technician put the parameter of gas mixtures, such as: name of components, conceptration and cylinder size, to the computer terminal, within 15 sec. all the technical data of the component properties such as: B.P., solidifying

point, sp,gr., toxic parameter, blending order, filling pressure temp., cylinder kind, marking, transportation code are displayed manifestively. the development of this set soft ware has important warranty about the quality, it promoted the working efficiency and blending precision.

(3) Research about blending method

The blending method common used internationally has two kinds:

the lst kind is static method including i) Gravimetric method ii)

partial pressure method iii) fixed vessel method iv) dilution method.

those companies in Western Europe used commonly the gravimetric method,

partial pressure method, flow ratio method and the synthetic application

about those methods stated above. a brief introduction is as follows:

a. Blending gas in using the Gravimetric method

The principle of this method is to use precise balance to weigh the weight of empty cylinder, component gas and cylinder after blending, and then convert the measure to % concentration. this method is suitable to compound the low concentration blending gas (ppm level), generally, it can obtain higher accuracy. It is the basic method used for the manyfacturing of standard gas internationally. at present, in Western European companies, it is aided by computer to operate the fundamental procedures:

- i) Based on the name, concentration of components, use the computer to give out the weight of componets and relative parameters,
 - ii) Take the evacuated cylinder, weigh it on the balance.
- iii) Fill the cylinder with component gas, and weigh again to the determined weight.
- iv) finally, fill the background gas to the total fixed weight.
 This kind gas mixture which is using the above method to produce

needs no detection, it can be transfered as a standard directly.

Errors in this method are mainly: i) balance error ii) process error iii) calculating error among which the balance error is key error, therefore, the balance precision is the main target in evaluating the blending gas method whether it is sophisticate or not.

Based on the blending gas precision and tolerance, the France air liquid CO. installed so many balances with different precision. In the Mitry-Mory plant, we saw the high precision balance in the blending gas shop and also the single arm mechanical balance and electronic balance with different precision. there was not any peculiar requirements about the balance circumstances.

It is reported, if you want to prepare standard gases with peculiar requiremnts, you must make buoyancy correction. the installation stated above is used only for preparing super precision grade gases. MG CO. of Western Germany provided with gas balances which could weigh to Max. weight 28Kg (sensitivity is 1 mg), that Co.requires the balance room which must be themostat and constant humidity.

The specialty gas plant of Linde CO. in Western Germany allocated precise balances (max. weighing weight is from 10Kg to 100Kg). it is said, the minimum preparing concentration of that plant can reach 10-100ppb. in the CREW specialty gas plant of England AP CO., we have seen a series precise balances, among which the max. wt. are 120Kg, 110Kg, 100Kg, 37Kg, 10Kg, respectively, the relative sensitivities are 1g, 10mg, 10mg, 1mg, and 10mg respectively. in the mean time, we have seen a set of secondary generation precise balance which was under trial produce, that installation didn't use the mechanical arm, it adopted base wt. plate support method. through the sensor to transfer to gyroscope and then through the single amplifier to display the weighted. data, it was introduced, as soon as the installation was manufactu

red successively. the max. weighing weight will be 200 Kg, the sensitivity can reach 2mg.

At present, the equipment level of common use large scale precise gas balance in our country almost approaches to foreign countries level, the disparities are mainly as follows: i) high precision electronic balances of different kind are not in popularization. ii) the research about next generation high precision balance is still in blank space.

- b. Blending gas by partial pressure method is: based on the gas pressure law, take two kinds or more kinds mixable gases, filling it according to different pressure into cylinders which are already exhausted. Since the computer soft ware developed, the blending precision of partial pressure method increased so much. at present, it is one of the important method to blend gases in western Europe. the operating procedures are as follows:
- i) Based on the name, concentration of the desired components, the computer gives out the partial pressure and relative parameters of different components.
- ii) Pill those component gases into the exhausted cylinder in series, until it reaches the pressure under regulated temp.
 - iii) Pinally, fill the background gas until the final pressure is reached.
- iv) After mingled homogeneously, use the corresponding method to detect the component concentration.

Errors of this method are mainly in:

- i) Deviation of the correct value of ideal gases.
- ii) Error of blending installation and the pressure gauge.
- iii) Operational errors.
- iv) Analytical errors.

The main reasons of error are the above first two items. since the computer

soft ware has been developed, the first problem has already been conquered, at present, those companies pay focus attention to the following two points:

i) research different kind of precise blending installations and high precise blending pressure gauge. in order to minimize the instrument error during blending at the max. limit. ii) transition from semi-automatic to fully automatic operation. the France air liquid CO. announced that the blending process would adopt wholy computer control, all the valves opened through the installation automatically, so that the systematic errors which were brought by labour operation could be conquered.

In the Prance air liquid CO., we investigated the blending shop of electronic gas of CHALON factory, that shop could compound arsine, silane, phosphine, borane, diclorosilane, trichloroboron, hydrochloric gas, high purity ammonia etc. the total number was nearly 35 electronic blending gases. in the shop room, there was divided to two rows which allocated 2 sets of plate type partial pressure blending cabinet. each set had high precise pressure gauge and precise valve. it could compound 4 kinds of blending gas. all of the backgroung gases which were through the stainless steel pipe entering into the blending board adopted the integrated supply gas method. among which, the supplying method of He, H, adopted the cylinder container type to supply gases in parallel. the supplying method of Ar, N, adopted the liquid state completely. All of the doping gases used cylinders which were filled in outdoor. in the shop, it was in negative pressure, the pressure difference in comparing with the outdoor pressure was 20Pa. before the operating board of each set blending installation, there was plastic canvas hanging curtain, it adopted laminar flow protection, the arrangement of the whole shop is reasonable, construction is compact, safety, clean, easily to operate. that plant adopted a method

which integrated the partial pressure method and gravimetric method for blending gas. In one blending installation, it could blend the same component and content gas mixture in several cylinders, during blending, put one cylinder on the electric balance directly to weigh the weight, using the weighing result to check different component content of gas mixture, and compared it with other cylinders, based on the method of blending, the once through successful rate is 100%, blending tolerance \$5%, this value compared with the analytical data made by CLAUDE IDELORME research center, the tolerance is not greater than 5%, it fully complianced with the blending requirements of electronic gases.

e. FLOW ratio method (also called as Dynamic flow quantity method)

The principle of this method is to control the flow quantity of

different component in gas mixture, so that, it can modify the flow ratio
to compound different gas mixtures.

This is one kind of newly developed industriallzed blending method. it is one kind of easily operating and high efficiency method for blending large batch gas mixture with the same component and same concentration. we have seen such installations in the companies of France, West Germany and England.

But from the view point of comparison, the flow ratio blending installation of Mitry-Mory of France air liquid CO. is the best. its blending procedures are as follows:

- i) Open the valve, let the component gas and backgroung gas pass the pipe under 13 atomospheres.
- ii) Pass the component gas through the thermostat region, in order to make the temperature equilibrium,
 - iii) Pass the component gas through supersonic flow cabinet, de-

termine its flow accurately.

- iv) Based on the blending insturction which is already inputed, the computer will select the blending orifice plate.
- v) Pass those component gases through the selected orifice plate to the storage tank for mixing.
 - vi) Pressurize the blending gas to 150Kg/cm2.
- vii) Pass through the manifocdo for filling. it is reported, in using this method, it can compound 20 cylinders of 4 element gas mixtures at one time. if the number of components are greater than 4, then, it can be compounded separately. the blending range is 2-5%, preparating precision can reach 1%.

From the blending technology of companies in western Europe, there is one important feature in using soft ware development and computer technology. it makes the traditional process having a fundamental modification which raises the working efficiency, minimizing the labor intensity and guarantees the product quality.

(III)Analytical detection technology

Those gas companies in western Europe pay more attention to the analysis and detection technologies. in each key point including from the gas material analysis, process detection, intermittent products control to produce quality examination, there are relative detecting instruments for monitoring. it forms a complete quality assurance system, so that the process can be modified, raising the product quality, promoting the production development. now, a brief introduction about the gas analysis gives in below:

1. The class of analytical appratus is complete, the feature is advance.

The specialty gas production plant of different gas companies in Western Europe usually allocated two class analytical laboratories:

one class is the synthetic laboratory which has sophisticated equipments with complete set of apparatus, it aims mainly for the synthetic research and experiment. the other class includes such special instruments located in site, it is used for on line monitor and product examination. a large portion of apparatus used in different companies is the products produced between the end of 70 centuries and the bebining of 80 centuries. the technical targets are more advance, most of them have micro-processor or adopts program control, use the electronic brain to form net work. Situations of the main equipments are as follows:

- (1) The chromatograph adopts many kind detectors, such as TCD, FID ECD, FPD, HID, AID, HWD etc. the constructions of the instruments are composed single detector, double detector or poly-detector. the chromatograph also adopts different fixed state monomer fixed and liquied therefore, it can make the analysis of different impurities content.
- (2) O₂ analyzer: the species are also in completeness, generally, it has the constant amount and trace amount analyzer which can use in laboratory or on line analysis.
- a. Constant amount analyzer: generally, it uses the magnetic O₂ analyzer for on line analysis, in the mean time, it also uses the chemical absorption method for analysis.
- b. Trace amount analyzer: it commonly uses the following 3 methods: solid electrolyte, different concentration battery method, Hertz cell
 method and primary cell method.
- (3) Trace amount H₂O analyzer: it commonly uses the following 4 methods: condenser method, Coulomb method, piezoelectric adsorption method and dew point method (rarely used). among which the piezoelectric adsorption method and Coulomb method used commonly for on line analysis. because its sensitivity is high, speed is fast, construction

is simple, easily to operate, the price is cheap. besides this, the Linde co. of western Germany is now researching the quarz detector to analyze the trace amount H2O content in 7 grade gas.

- (4) Infra-red analyzer: it has 3 kinds:
- a. Infra-red spectrophotometer: it is commonly used in the qualitative and quantitative analysis of gas mixtures. Sampling at one time can determine the impurities content of several or more than 10 components.
- b. Infra-red gas analyzer: it is commonly used for the on-line analysis about mono-component, such as to determine the trace amount CO, CO2, NHO in gases. this kind instrument has high sensitivity, quick response, read directly. it is an ideal on line analyzer.
- c. Fourier transform infra-fed spectrophotometer: this instrument is an ideal apparatus for the qualitative and quantitative analysis of poly-component impurities in gas mixtures. for instance, AP CO. of England bought a 1750 type FT-IR from PE CO. (USA), they use it to do research work for analyzing trace amount CO, CO₂, CH₄, and C H in six grade gas. the min. detection amount < 0.5ppm. the air liquid research center of France and Linde CO. of Germany bought 60 sx type and SDXB type FT spectrophotometer manufactured by Nicolet CO. respectively. such instruments can use with gas chromatography parallely, therefore, it can obtain the optimum separating effect.
- (5) Atomic absorption spectrometer: it is used to detect the metal impurities in the electronic grade gas, because the main impurities are alkali metal and heavy metals. especially, in the semi-conductor industry, the metal impurities must be analyzed. based on the user's requirements, those companies do the metal impurities detection by using advanced instruments, such as: AA3030 type of America PE CO., AA40

type atomic absorption spectrometer of America Varian CO.. Instruments stated above must use peculiar gas sampler, in order to complete the gas analysis tasks.

- (6) Nitrous oxide analyzer: it is used for analyzing the nitrous oxide in gases and O₂ for medical use. they commonly select those instruments produced by America thermo-electronic company and Backmann CO. the principle used is "Chemical Luminescence method".
- (7) Mass spectrometer: this kind instrument is the important analytical measure used for those CO. in Western Europe. it has single focusing mass spectrometer and poly-detector in parallel, for example GC and IA used parallely, it can raise the analytical function for detection those complex items. recently, it can raise the analytical function for detection those complex items. recently, so many companies bought the international most advance MS, such as Type 8-80 MS of England VG CO. Type 5100, Type 271/45, Type 251 MS of US MS CO. etc. The instruments stated above can do the work for analyzing the low mass number impurities in gas and isotopes.
 - (8) Other peculiar specialized made instruments:
- a. Photo ionized detector (PID):used for deteting AsH₃, PH₃, NH₃, and C_H analysis. (Made in England BOC)
- b. Spectrum analyzer: used for detecting N₂ in Ar, Type MK2. detecting range: 0-100ppm (Made in England BOC)
- c. AsH₃/PH₃ analyzer: type 106 detector, it is a high temp. PID, sensitivity is 20ppb. this instrument commonly used for electronic gas shop alarming: alarming concentration: AsH₃ 50ppb, PH₃ 300ppb (Made in England BOC)
- d. Coulomb electrolysis type H₂O analyzer: Type W, measurement range O-10ppm H₂O, read directly. (Made in England BOC)
- e. Nitrous oxide analyzer: type L201, measurement range:o-10ppm (Made in England BOC)

f.CO analyzer: use the chromatograph method: thermo-condutivity detector, measurement range: 0-50% CO, used on line analysis. (Made in Germany LINDE CO.)

- g. Gas phase spectrometer IIP-1000 type. thermo-conductivity detector, used for analyzing trace impurities(ppm) in gas. (Made in Germany LINDECO.)
- h. Trace 0_2 analyzer: LMOS-IVR type, primary cell method. measurement range 0-10ppm, 0-40ppm 0_2 . (Made in Germany LInde CO.)
- i. Spectrometer: luminiscence spectrometer method: analyzer of N₂
 (trace amount) in Ar. lowest detecting quantity N₂<0.5ppm (Made in Germany MG CO.)

(9) Ultra-violet spectrophotometer:

It is use for analyzing the impuriries such as SO₂, H₂S. the lowest detection limit is lppm. sampling 5ml(PE CO. of USA) at present, Linde CO. researches to use the ultra-spectrophotometer to determine analytical items which can't be solved by infra-red spectrometer, it earns big strides.

2. Analytical research goes deep into everyday, the detection limit gets lower and lower. the specialty gases department of France air liquid Co. goes to research and develop the analytical technology about those components lower than ppm grade. formerly, for instance, the Silane used in semi-conductor diffusion process must have the ppb grade, they use the traditional electro-resistivity method to analyze it, but it can't reach the requirements of detection. now, in using spectroscope method to determine it, the analytical precision raises greatly, and also, for the analytical detection about scientific material, a new analytical method is now under development, the special analytical instruments have done well about the preparatory work for the production plant.

The research department of Linde CO. in Germany used the 7 grasde gas as the carrier gas, adopted the gas-mass chromatography to detect M_2 , O_2 and N_2 in sample, it obtained fruitful results.

Determination of humidity used the quartze detector, they are now researching to use ultra-violet spectrophotometer to determine trace amount sulfide in gases. this hardly can't be done by infra-red chromatography, and even it was not reported in literatures, besides this, the above companies develop a batch of specialized instruments which have high sensivity, simple construction, easily to operate, strong directly perceived through the senses and low price. it obtains good evaluation of process when it is used on line analysis.

3. Pay attention to the research and development of the method Sampling is the key point for the detection of gas, liquid state product. under any circumstances, sampling always has errors, we want to minimize the error. source of errors are: i) diameters and capacities of package vessels are different, the gas quality in the internal part is non-homogenous, it has separating layer phenomenon. ii) leakage of air. because after gas flow be expelled out to air, it exhibits anti-diffusion phenomena. iii) the variation of flow velocity leads non-homogenity, hence, to select suitable sampler is the important warranty for minimizing tolerance and raising analytical precision.

The sampler designed by Linde CO. of Western Germany, first of all, adopts sampling under low temp. afterwards, raise the pressure by means of vaporization, this kind sampling prevents the 3 factors influence stated above. gurantees the precision and reliability of the analytical results.

Besides this, they research a soft package sampler with antipermeable features, low pressure atandard gas aluminum cylinder and the special gas sampler used in atomic absorption spectrography. all of those stated above raise the analytical precision, gurantee the analytical quality.

4. Lay stress on the routine contrast work for standard gases The measure transfer base of the calibration gas for instruments and meters used in the 3 countries of Western Europe are taking the NBS (USA) standard gas as the base. those detection method and instrument installation are also taking the NBS code as the basic foundation. it adopts many methods for contrast the blending method for calibration gas, usually, it adopts gravimetric method, such as the England 0, CO. selects the balance with max. weighing wt. 66kg, sensitivity 0.5-1mg; max. weighing wt. 200kg, sensitivity 0.2g. the blending gas precision: absolute error 2%(lppm grade). blending precision: absolute error 0.01-0.02%, relative error: 2% (ppm grade) Air liquid CO. of France selected such balance: max. weighing wt.100Kg, sensitivity 10mg, blending precision in ppm grade, relative error 2%. (the standard produced by that CO. is approved by NBS . of USA). MG CO. of Western Germany selected a balance with the max. weighing Wt. 100Kg, sensitivity 100mg, and max. weighing wt. 25Kg, sensitivity lmg, blending precision in ppm grade, relative error 2%.

The 3 organizations including BOC CO. of England, NBS of USA and national physical laboratory made contrast periodically, they also contrasted with other countries of Western Europe, but standard gases produced by BOP of Englandwere still not approved by NBS of USA.

NBS of USA approved normally that air liquid CO. of France is a standard gas producer, but they are still to make contrast with other countries of Western Europe, and also contrast with laboratories of the CO. itself the relative error is not greater than 0.5

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In Linde CO. of Western Germany, the standard transfer is using the 2nd grade SRM supplied by European standard association as the standard, and SUM is through the measure of NBS of USA to be transfered.

From the summary stated above, the standard for measure transfer of standard gases in the 3 countries of Western Europe and NBS of USA are generally recognized by different countries as the "absolute standard."

5. Analytical instruments

Synthesize all of those analytical instruments used by 3 countries, in according to the kind, characteristic, usage and manufacturing plant, enumerate a "Schedule of analytical instruments". see annex.

(IV) Package and transportation technology

Recently, in accompanying with the new technology development of electron. light fibre, standard measurement etc. it promotes the specialty gases to have a synthetic development trend which are polyspecies, series, high purity, multi-component and low concentration, in order to accomdate the requirements of such development, to guarantee that specialty gas having the good characteristics of high precision, high stability, high reliability, they made new modofications for package and transportation technology.

- 1. Package in gas state
- (1) Renew the cylinder mater is continuously

In order to increase the strength, decrease the wall thickness, mininize the weigi. ncrease ductility and durability of gas cylinder and accomodate the requirements of poly species of gases, those companies in western Europe are now urgent to find new cyninder materials.

Take the high pressure seamless cylinder as an example, since half centuries, it has changed the materials from low carbon steel (0.2% C), medium carbon steel (0.4% C) to manganese steel (0.35% Cr, 1.5% Mn). at present those western industralized countries are commonly adoping Cr-Mo steel (0.35% Cr, 1% Mo), Al-alloy steel and other peculiar alloy materials.

In our country, the large portion of material of cylinder selects the Mn steel, it has much shotcomings in the fields of tensile strength, yielding strength, cylinder wt, gas capacity in comparing with foreign cylinder, therefore, to quick develop the steel species and heat treatment process are the main problems for the development of cylinder.

(2) Many specifications of gas cylinder has been developed

There are two kinds of gas packageing which are rigid vessel and nonrigid vessel, in accompanying with the development of transportation in liquid state, the demand of large gas cylinder decreased continuously. however, the demand of small gas cylinder increased so much, such as England AP CO. used 9 species of high purity gas cylinders which are 0.37;2;8;9;9.5;31.3;41.2;43.5;108.5. besides this, it is more fastidious in thoe respects about shape design, outlook quality, cylinder decoration, such as, France air liquid CO. made pink plastic mesh decoration on the shell middle part of gas cylinder, it seemed very beautiful.

In the field of non-rigid vessel, recently, those companies developed different kind plastic seal soft package vessel mede by high layer compound membrane. it used mainly to store different gas, mixtures. its advantages are: easily to make component stable, price cheap. we have seen a series of plastic bag made by poly-ethylene and Al-compound membrane in Linde specialty gas, its capacities usually in 1-50 liter. filling pressure 790-810mm H₂O, optimistic concentration range for storing standard gas is 10 ppm-5%. It is reported, this kind plastic bag can be filled with pure gas, ppm grade propane, n-butane and mixture of C H with N₂, within a period of 7 weeks, the change of gas mixture concentration is not greater than lppm. fundamentally, it satisfies the requirement of gases used in calibration of instrumments.

(3) Technology to treat inner wall of the gas cylinder has new development

Cylinders used for packageing super high purity gas and gas mixture of low concentration, standard electronic gas mixture cylinder must take severe inner wall treatment. otherwise, it will influence the product quality and stability of components. What are the reasons

to form the circumstances stated above? i) gas components adsorbed by iron rust in cylinder ii) gas components dossolved in remaining water in the vessel iii) gas components react with the inner wall of vessel iv) gas components have adsorption or desorption effect on the vessel inner wall. in order to expel such interfering factors, long stablished to store different kind stanard gas mixture and electronic gas mixture, gas companies in Western Europe developed their inner wall treatment technologies of gas cylinder. it is reporteds at present, there are 4 methods:

- a. Gold plating method: in the inner wall of vessel plated with gold, so that to isolate the contact between the inner wall and gas component, to keep the gas stability, this method has valuable effect in storing low concentration standard gas mixtures. all of those gas companies in England, France, Western Germany hold this technology.
- b. Do the pretreatment by filling those blending component gases: use high concentration blending reacted gas to do the pretreatment for gas cylinder, so that the inner wall will be adsorbed saturately and then refilling of the required gas. such as AP CO. of England used this method to treat electronic gas mixtures.
- c. Pretreatment with filling oxidized gas: after the cylinder to be treated normally, fill the oxidized gas to make the innor wall as a stabilized oxidizing membrane, in order to minimize the desorption and adsorption effect of inner wall, the oxidizing gas can be dry air, O_3 , NO_2 or inert gas mixture which has any optical proportional O_2 content, In the MG CO. of western Germany, we have seen them treating the electronic gas mixture cylinder in using this method.
- d. Painting on the inner wall: On the inner wall of gas cylinder painted with such organic materials of wax or fluro-resins. in

order to isolate the contact between component gas and vessel inner wall. In Mitry-Mory specialty gas plant has a complete gas cylinder treatment system. the prodedures are: i) Inner wall blasting, using \$0.3 artificial glass balls ii) Examine the rust spot by means of light conductivity fibre ii) Ultrasonic washing by means of F1135 washing agent iv) Organic material painting by means of painting ma chine (Painting formula is know-how). v) Install the cylinder mouth vi) Spray lacquer about the gas cylinder outer wall and dry vii) drying by means of heating and evacuating viii) gas filling. it is reported, cylinders containing AsH₃treated by this method can be protected its stability of ½ year, for storing SiH₄ gas mixture, its stability can be protected 1-1.5 year. the stability of Borane is

- 2. Packageing under liquid state
- (1) The species of low temp. vessel has a trend to be serialized day by day

The species and warranty of low temp. vessel in western Europe has a trend of rapid increasing, such as, from the general adiabatic vessel for transporting natural gas to vaccum adiabatic vassel, vaccum powder adiabatic vessel and high vaccum fibrous adiabatic vessel for transportating liquid $\mathbf{0}_2$, liquid \mathbf{N}_2 , liquid Ar and also using big railroad and tank cars. Those companies have their serialized products, during investigation, we have seen small size Dewar bottle with a capacity liter used in laboratory, and the max. liquid $\mathbf{0}_2$ vessel with a capacity of 5000 tons, this situation reflects the large demand of liquid transportation from other side.

(2) The transportation technology about non-condensing gas goes successively day by day

At present, those western European countries adopts liquid transportation about H₂, He, low B.P. non-condensing gases. the high vaccum multilayer adiabatic vessel, special vessel with kiquid N₂ curtain or conducting curtain and transportation tank truck have already formed its industrial production scale. In MC CO. of western Germany and England gas and equipment Ltd. we have seen the large highway tanktruck which has a length of 20M with a capacity 11.3M³, by estimating, the filling capacity of that tank truck is nearly 3 times about the production capacity of our plants.

- 3. Transportation fashion
 - (1) Rapid development about liquid transportation

Because transportation in liquid state has the advantages of low contamination, large capacity, low freight fee, low labour intensity, therefore, recently, in Western Europe, they almost adopt liquid transportation about large amount gases as H₂,Ar,He, O₂, N₂ etc. this trend promotes centralized the gas industry, such as the France gas market is almost momopolized by Air liquid CO.

(2) In Western Europe, at present, the traditional scattered load and unload fashion is replaced by the gas container, we have seen, no matter, in the cylinder warehouse or during transportation, all of the gas cylinders which made by 8-12 cylinders as a group put in a container made by iron wire fence and the load and unload are done by fork truck.

The container divided to vertical and horizontal type, and it usually adopts the filling and supplying gas method in outdoor. in these countries, at present, it seldom sees the load the and unload by human labour.

(3) Pipe transportation about industralized gased are very florishing

In the Ruhr industralized zone of Western Germany, there is

pneumatic pipe net work about 16000H³/hr, the radius for supplying gas is 120KH. this is rarely happened even in developed countries. this pipe net has an advanced design, resonable arrangements and enough to meet need, severe management system. since sypplying gas 7 years, any accident didn't happen.

(V) Safety protecton technology

Electronic safety protection and detoxicate technology are important problems in safety technology. the CHALON electronic gas plant
of France air liquid CO. solved well about these problems. the permissible concentration of toxic gases in that plant shop obeyed completely
the OSHA regulations of USA (it belongs under the managements of IGC
whose headquater is in Paris). the specified targets are as follows:

Schedule of permissible concentration in toxic room

N.B. TWA means time waited average

Schedule of expelling concentration in toxic room outdoor

Through different positions of the room to monitor the concentration of toxic gas indoor, in regarding the outdoor concentration of toxic gas, it is also monitored and alarmed by automatic monitoring instruments in different positions,

Window of shop must be sealed severely, to keep indoor pressure less than outdoor 20 Pa. the roof is light, all of the electricity equipments must be explosion proof.

The remaining toxic gas returned from users are burned with propane, the chimney is 25M height, but the SiO_2 and Si_2O_3 powder obtained from the combustion of $Sill_A$ must be filtered out and recovery together.

The treatment method for Arsine in that plant is, first of all, heating the arsine to produce the penta or trivalent oxide of arsine, and then, put those oxides in CuSO₄ solution to absorb it and transform it to copper arsenide, heat copper arsinide to produce Cuo and As₂O₃. finally, use alkaline solution to extract the oxides of arsine to form white arsine crystals, recovery and use it.

In MG CO. of Western Germany, we have seen the research work about recovery waste gas of Arsine. they adopt two methods: first method is just as stated above, under 300°C, burnt the AsH₃ and react it with CuSO₄ solution to form CuAsH₃ and then absorbed by Na₂CO₃. the 2nd method adopted changing colour agent which adsorbed the A₂H₃, through the change colour degree of adsorbent, it showed the time necessary to change adsorbent (the saturated adsorbent must be recovered by relative factory to treat it again).

Those comapanies in Western Germany which produce toxic gas mixtures take the following methods for the safety drain and recovery of its waste gas:

1.
$$SiH_4$$

 $SiH_4 + 2H_2O \rightarrow SiO_4 + 4H_2$
 $SiO_2 + 2KOH \rightarrow K_2SiO_3 + H_2O$ (Solution of KOH is 47-107)
2. B_2H_6
 $B_2H_6 + 6H_2O \rightarrow 2H_3BO_3 \rightarrow 6H_2O$
 $2H_3BO_3 + 6KOH \rightarrow 2K_2BO_3 + 6H_2O$
3. HCI
 $HCI + NaOH \rightarrow NaCI + H_2O$
4. PH_3
 $PH_3 + 8CuSO_4 + H_2O \rightarrow H_3PO_4 + 4H_2SO_4 + 4CuSO_4$
5. SiH_2CI_2
 $SiH_2CI_2 + KOH + H_2O \rightarrow KSIO_2 + HCI$
6. GeH_4
 $GeH_4 + 2H_2O \rightarrow GeO_2 + 4H_2$
 $GeO_2 + 2KOH \rightarrow K_2GeO_3 + H_2O$

The new trends of applied technoligy in western Europe are mainly expressed in the following respects:

(VI) Applied technology

1. Applied service system is more completed cntinuously

Those countries in western Europe pay more attention to applied service work. in accompanying with any one kind new gas product which is coming out, then those accompanied applied equipments, such as, compounding gas cabinet, on line instrument, pressure regulator, flow meter, special pipe fittings, valves etc. are linking up the parts to form a whole set supplying to customers. those gas plants can deliver gas to the doorstep or established a special supplying gas station for facilitating the usage of gases.

Besides this, different large companies eatablished applied technical service center and emergency response, emergency squad by themself. they give service within the whole day. for instance, any user has found some equipment trouble, safety accident or technical problems, he can contact with the service center instantaneously, and the technician will soon go to the site for technical information, solving trouble or dealing with the aftermath of the accident. the user's service center of Prance air liquid CO. has branches in several places in order to guarantee that any user has problems which will be solved within one hour.

2. Directing thought of applied service is just in modifying

At present, the technical servicing work of those compaies in western Burope has been changed from passive position to initiative position. such as the research center of Prance air liquid CO. established electronic gas research section, laser research section, in which there are persons specialized assigned for the research task about new technology and relative parts of gases. In order to sale the liquid N₂ used widely in the field of food quick frozen, low temperature storage, rubber drawing of patterns and de-rim repair under freezing pipe, MG CO. of western Germany estabilished special equipment plant

1-1

which was producing different kind of quick frozen machine, medical low temperature cold storage, rubber drawing pattern machine and pipe quick forzen machine etc., and they supplied to users such equipments with liquid N₂ together, MG CO. considered such methods for promoting to sale goods are an effective measure which can attract users tap market and prevent users transfering to other salers.

3. Development new applications about traditional products continuously

The England distiller CO. is a small CO. speciallized to sale ${\rm CO}_2$, but they obtained manifest success through developing new applications about ${\rm CO}_2$. for example:

(1) Greenhouse plantation process using rich CO2

Owing to the research work, they discovered that plants lived in the rich ${\rm CO}_2$ atomosphere were easily to quick to photosyntes is action, shorten the production period, increasing the product quantity. therefore, they develope this technology to fill rich ${\rm CO}_2$ which had a concentration about 1000ppm ${\rm CO}_2$ to the greenhouse. the result was producing a big harvest area about tomatos, cucumbers, bambooo shoots, vegetables etc. in short period. the consumation of ${\rm CO}_2$ in summer is 2.5T/acre, in winter 3.5T/acre, this advanced technology which was transfered to other districts made the sales amount of ${\rm CO}_2$ increased enormously.

(2) Dry ice expansion technology of tobacco

Through the research work, that CO. discovered the following phenomenon, during the processing of tobacco, if the tobacco was packed in liquid CO_2 , the Nicotine would dissolve in liquid Co_2 and then to be freezed to dry ice, after ards, vaporized it through high temperature. large amount nicotine could be brought out by means of vaporization, in the mean time, the surface of tobacco expaned.

more than one time of the original capacity, in this case, not only it decreases the harmful efcect of tobacco, but also increasing the production capacity, that CO. made joint ventres with tobacco salers established 200 new cigaratte plants in 1983, each production line could consume CO₂ 880Kg/hr. owing to the development of this new technology, it earned high profit and broadened the CO₂ sales amount.

(3) The CO, share filling technology in beer

Beer is a kind beverage which is welcomed by whole world. but in bulk beer, the CO₂ content is easily to be spreading out. finally, the rmaining lacquer lost its foam and its original flavour. that CO. suggested a simple and easily to operate technology, first of all, used the cylinder CO₂ to fill it in the barrel, then the beer flowed out of the barrel by aiding of the CO₂ for share filling, such share filling beer remained its rich foam permanently, and also the colour was transparent, taste good, warm Welcomed by those consumer.

(4) Low temperature extraction technology of CO₂

Because in the traditional extraction process used organic solvents which contained Cl⁻. it is forbidden to use such solvents in western countries, under such conditions, based on the characteristics of CO₂ which are non combustible, non-toxic, cheap price, easily to buy they developed the new technology using the critical state CO₂, to extract different kind organic substances, such as, extract coffein from tea leaves, extract perfume from lemon grass, extract carotene from carrots, extract ginseng spirit from ginseng, in usig this method to extract, it couldn't change the components of extracted substances and its properties were very near to the natural compounds.

From the practical experience of CO₂ CO. the development of product applications has no limit even in the field of traditional products

there are still many unknowns waited us to explore.

III. the development situation about main species of specialty gas in western Europe

Applications of specialty gas have widely used. species of it are enormous, at present, most research power put in this field in western Europe. there are 4 main species of specialty gases which have the largest production capacity.

(I) Electronic gases

We call all of those specialty gases used in electromic industry as electronic gases. recently, in accompaning with the development of micro-electronic technology, the species and quantity of electronic gas increased enormously. requirements for its quality are more and more higher. through understanding, at present, the 64K large integrated circuit can be produced already industrially in western European countries, the 256K super large :integrated circuit is now entering the industrial production stage. in 1984. the total producion capacity of large integrated circuit of the world was more than 450 hundreds of millon plates. at present, those countries concentrate their research power to tackle the technical problems of 1M-4MVLSI. In front of the rapid growth of market, the western European countries are now finding the right technological decision positively, in order to grasp firmly the development and preparation about new process and new technology of electronic gases.

The species of electronic gas are more and more abundant
 The integrated circuit is a kind enterprise which renew and change generation fastly, since 60 centuries, the integrated circuits used in 60 centuries were now not suitable to use in 70 centuries,

In accompanying with the discovery of new technologies such as, ioninjection, low temperature, low pressure, erosion engraving (dry method). those companyies developed such new products, such as, SiH_2Cl_2 : CF_4 , CF_6 , CHF_3 continuously which replaced the first generation erosion engraving agent such as FH, $SiHCl_3$, $SiCl_4$, old products. in order to accomodate the necessary of 80 centuries for the new technology to proces the line width < lam, the third errosion engraving agents of NF_3 , MOF_6 , WF_5 are developed. in order to accomodate the necessary of chemical precipitation super thin membrane, the new technology of MOVCD needs different kind metal organic compounds which are developed continuously. such as France air liquid CO. has developed already $Ca(CH_3)_3$, $Al(CH_3)_3$, $Al(CH_5)_3$. $Te(CH_3)_2$, $Sb(CH_3)_3$, $In(C_2H_5)_3$. MG CO. of western Germany developed $Ca(C_2H_5)_3$, $In(CH_3)_2$, $Cd(CH_3)_2$, $Cd(C_2H_5)_2$, from the statistical view, the species of electronic gases are about 115 in the whole world.

2. The purity of electronic gas is more and more higher

Because the configuration and process of super large integrated circuit

have severe requirements to its precision and fine distinction

particularyly, those gases which are used for producing semi-conductor

crystals must depreivation the harmful elements of III group and V

group of elements in periodic table below ppb grade. therefore, the purity

of electronic gas is very important. in order to satisfy this conditions, MG CO.

of western Germany has tackle the key problems about 7 grade gas (99.9999%).

Total impuritie content < 0.1ppm. the trace amount is the limit for detecting

instrument. at present, they develop three 7 grade species, namely, Ar, He,

H2', the purity of SiH4 has also reached 99.9999%, they also developed the packing

vessel for high purity gas and transportation gas. it is reported, that CO.

is the

rid;

sole plant which used steel cylinders to transport 7 grade gases.

- 3. The limitation of dust particles in electronic gas is severly restricted In the super large integrated circuit above 64K, the line width of it has reached 2.54m. such processing, not only subject to special requirements to the purity of gases, but also to control the dust particles in gases, those electron companies have an agreement that the max. dust particles can not be greater than 0.254m for 64K circuit, 0.154m for 256K circuit. in order to satisfy such new requirements, those gas companies developed the ultra-clean, zero contamination, terminal filter installed in the purification gas line. it is reported, at present, the high efficiency filter which can filter out the 0.14m dust particles can be manufactured, but the detecting technology for trace amount dust particles in super purity gas is not up to standard in European countries, the characteristics of filter are checked indirectly through examining the electric circuit feature generally. In Prance air liquid CO, and western Germany MG CO, laboratory, we have seen their technical persons doing the research work about dust particle detection.
- 4. Requirements of sensitive metal impurities in electronic gas are more and more lower

Some metal impurities in high purity gas, such as K, Na alkaline metals and heavy metal transcendental metals have important influence to the electricity properties of .integrated circuit. this kind influence becomes more sensitive in accompanying with the integrated degree raises quickly. therefore, in the production of super large integrated circuit, not only there are severe restrictons about the

impurities content in ultra-pure and chemical reagent, but also there are some requirements for high purity gases. at present, the western European countries now endeavoured to develop the electronic technology about metal ions in high purity gas, In the France air liquid CO., MG CO. of western Germany, Linde CO. and AP CO. of England, we have seen they do research work by means of atomic absorption spectrophotometer instruments, but how do control the harm ful metal inpurities in high purity gas, there isn't any effective method now.

5 Special complete set parts for electronic gas are more and more integrated

Electronic gas is an important branch of specialty gases, because the species of electronic gas are enormous, features are complex, technology is fine, therefore, the application problem is also conspicuous. in order to stabilize the supply for electronic gases to those users, the western European countries pay attention to the development of complete set parts of electronic gases, recently, Prance air liquid CO. and western Germany MG CO. develop a series package: vessel for electronic gas, cylinder valves, regulators, filters, flow meter, alarming installation, cleaning installation, detoxicate installation and cleaning gas line etc.

We call those specialty gases used in medicine as medical gases, at present, Species of medical gases developed internationally are O_2 , N_2 , N_2 , N_3 , N_4 , N_4 , N_5 , N_4 , N_5 , N_6 , ehtylene oxide and its gas mixtures, those gases used in the medical field, such as: diagnose, cure, anaesthesia, sterilize and clinical examination, playing more and more important actions, for instance, based on the components and concentration of patient exhale gas, compared it with standard gas, the doctor can deduse correctly some pathology factors, use O_2 /Ne gas mixture which can minimize the shortage of O_2 disease of asthma patients, promote the respiration activities recovering to normal condition, use liquid N_2 mixture to cure architis and rheumatism etc. at present, the France air liquid CO, and western Germany Linde CO, estalished specialty medical gas research department, they do large amount research works about the development and applications of medical gases.

Recently, the important products have the following species:

1. N₂O anaesthesia agent

The requirements of anaesthesia agent are that when dosing quantity charged in the short period, it will have clear effective change, the anaesthesia methods are: injection method, accupuncture method, and inhale method, at apresent, in western Countries, they adopt inhaleing method using anaesthesia agents as: cyclo-propane, trichlro-methane and ether etc. but after applying in clinical operation, there happened side effect, therefore, it is forbidden to use now. Linde CO. of wester armany has discovered that the mixture of N₂O+O₂ is a good inhaling anaesthesia agent there isn't any side effect during operation. Constituting the results of the case, 32N₂O+707-0₂ can be used in analgesic error for denotery, N₂O 507+5070₂ can be used in no pain parturition

FOR WOMEN; 80% N₂O + 20%O₂ can be used in analgesic effect for minpr surgical operation; 80% N₂O+20%O₂+small amount ther halogenide can be used for anaesthesia of the whole body. At present, Linde CO. of western Germany treats the N₂O production with severe quality control in according to the pharmecopia code of western Gernmany and produced also a series of applied equipments and apparatus used in the field of medical anaesthesia.

2. Ethylene oxide sterilizing gases.

In medicine, the commonly used sterilizing methods are hot sterilizing and cold sterilizing. the high temperature sterilizing belongs to hot sterilizing which consumes high energy. the sterilizing condition are restricted. the ultra-violet or ionized radiation belongs to cold sterilizing but its strelizing depth has limitations. recently, in western Europe, they develoe a kind safety cold disinfectant—ethylene oxide as sterilizing gas which has a mixing prescription of 10-15% C₂H₄O+CO₂ 85-90%, or 12% C₂H₄O+88%P₁₂, the advantages of this kind disinfectant is low temperature (55°C), it can, not only make no damage to non-water proof, non-heat proof materials, but also can make the clearance of the waited sterilized substance to be sterilized at present, it is widely used for sterilizing in operatioal room, clothes, sheet and various kind medical instruments. during sterilizing operatopn, it must be noted that, sterilize it in somewhat a prole ed time, in order to promote the remainder vaporized out.

In dealing with the ethylene oxide sterilizing gases, there are different views on this question, for instance, American experts considered that the remainder of ethyleme oxide contains carcinogenic substance which can make barenness of male, but such conjecture has not been proved, it is reported, the American industrial health organization makes relative restrictions about the content of ethylene oxide

in sterilizing gaa mixture.

(III) Food preserving gases

Those specialty gases used in the food industry so called food preserving gases which includes N₂,CO₂, ethylene and other gas mixtures, liquified products. it is an important trend in western Europe countries to used gases for preserving and refreshing of food.

Through the reserach work, those animal feature and plant feature food including meat, fish, egg, vegetable, fruit etc good culture medium for microorganism. if those foods stated above ...posed in air under room temperature, then it creates good conditions for the growth of microorganism under the catalytic action of enzyme. they will have rapid biological chemical reaction and become putrid. besides this, grease contained in food can have oxidizing reaction with 02 of air, the grease will be putrided and give rancid odor. Vitamin in food is also easily to be oxidized in air to form dehydrogen vitamin which exists no physiological function. the color of food may also happen to be changed by oxidation and lose its original quality. therefore, 02 is the important external factor for deterioration of food. Strictly control the 02 content in food preserving atomosphere is the basic condition to prevent food deterioration.

Vegetables and fruits, after picked, can preserve its living physiological function through respiration to make metabolism. but owing to lose the replenshing sources of water and nutrition matters for long period storage, it will become wither, colour changed, softening, weight decreasing, Vitamin decresing and finnally lose its freshness. In order to control the respiration of vegetables and fruits, it must control the O₂ content in food environmental atmosphere strictly, the food refreshing gas is just developed out based on the above tated principle, at present, so many desin-

FECTANTS, REFRESHING METHODS ARE ADOPTED IN FOREIGN CONTRIES. AMONG which the following 4 methods are related to gases:

1. Strorage method by adjusting gas quantity

Under certain conditions, in the definite temperature range (2-4°C), to practice the refreshing aim by means of decreasing O₂ content, increasing CO₂ content, the preserving atmosphere is 570₂+37CO₂+927N₂. this method is mainly used in the storage of large quantity vegetables and fruits. At present, the gas adjusting method is very popular in western European countries. it is reported, in France, the proportion using gas adjusting method for storaging fruit is 407, in England 80%. the large gas adjusting storage size has developed to the capacity of 10000 tons in foreign countries. our country has established the first gas adjusting stroage with a capacity of 28 tons.

2. Quick frozen storage method

This is a new method developed in the food low temperature storage process. recently, first of all. such method adopts liquified N₂ to make quick frozen about chicken, fish, shrimp, and meat. then afterwards, package storing. the ice crystals in the food are small and fine, so that the food tissue will not be destroyed. after defreezing, it will soon recuperate to the natural condition and not lose its original quality. this method was first developed by USA in 1960, and soon popularizing to western countries, we have seen in MG CO. of western Germany the plant which produce the quick frozen installations including immerse type, sprinlling type. cold gas circulating type installations. in USA, they firstly found to use CO₂ straight tube type and spiral type refrigerator for refreshing the fruit, chicken slice, Hanmburg bread with fruitful results.

Because CO₂ is cheap than liquid N₂, this is the reason for rapid spread of CO₂ in those countries.

IT WAS TRANSFEREND TO European countries in 1979, first of all, in western Germany, they developed a batch of refrigerator which entered to England market in 1984. we have seen, in England distiller CO₂ CO., the brief introduction about such fruitful results.

3. Low pressure storage method for vegetables and fruits
This method is, under serious decreasing pressure control,
to release the ethylene, CO₂ which are not suitable for storage.
in this case, the goal of refreshment will be reached, but we heard
only from the introduction about this method, we didn't see the
real installation.

4. Filling gas package storage method for food preserving International current food package has 3 types: shrinkage package, vaccum package and filling gas package. the fundamental principle of the 3 methods is to take food isolating to contact with 0, of air. in such case, we can obtain the refreshing aim in long period storage. shrinkage package is to sprinkle the food bag which is already exhausted, mouth sealed, in hot water or hot wind, so that the thin membrane contracted to wrap the food closely. Vacuum package is to exhaust all the air in the bag, such as various kind canned foods. filling gas package is a new package type which is developed in foreign countries recently, this kind method is putting the food in a well hermetic thin membrane bag, seal the mouth and exhaust total air, then, based on the food kind, fill in the bag with different prescription of refreshing gas so that the food will not change its quality, this kind package has manifest effect than shrinkage package and vacuum package. It is an ideal new package method. By introduction, vegetables and fruits packaged in filling gas package can be well stored about 1-8 month under room temperature. recently, Gas companies in western Europe develop a series filling package gas which are N_2 , CO_2 chemical gas and various proportion gas mixtures France air liquid CO. developed already a

SERIES FILLING PACKAGE GAS ES MORE THAN 60 species. each prescription has its own number. that CO. regulated a more detail applying rules for customers. such as, they regulated clear applying number, storing temp. and storing time for pastries, candies, meats (duck, goose, chicken, pig, ox etc.) vegetables, dairy products, seafoods (fish, shrimp, crab, clam etc.) beverages, complex ready to cook vegetables, so that the colour, taste, quality of food can be guaranteed.

(IV) Environmental gases

In order to detect the contents of contaminant substances in air, it must prepare different standard gas mixture of harmful gaseous substances, such as Cl_2 , NH_3 , HCN, H_2S , COS, SO_2 , NO, NO_2 , $\text{C}_3^{\text{H}}_8$, $\text{C}_6^{\text{H}}_{14}$, phosgene, chloro-ethylene, sulfo-methonal etc.

The England technical association selects more than 200 kind calibration gases mixtures to satisfy the requirements of various kind detecting instruments. those standard gas mixtures are so called environmental protection gas.

In accompaning with the development of industry, environmental contaminants in urban district is serious day by day. particularly the emmission substances expelled from fire power station, chemical industry zone and automobile tail gas, gave serious imperilling to the human environment. At present, industrial contaminant sources are many kinds, such as, oxysulfide comes from plant fixed growing source. CO, NOX, IIC come from automobile moved growing source (among which NOX is a high temp. product, generally, it is produced in highway. in our country, it has not yet the production condition now.) the US environmental protection bureau considered that if the 4 contaminants SO₂ CO. NOX IIC are to be controlled, then the other components will be ready solveed therefore, the research work of environmental contaminant concentrate in the control and detection of 4 substances SO₂, CO, NOX, IIC. our country's environmental protection

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bureau enacted "Atmosphere environmental quality standard" (see table II) in 1932. "Expelling standard of contaminants in high speed from automobiles" (see table IV) in 1983. In USA, they regulated the contents of automobile waste gas as follows (in 1968): CO1.5—2.5%, HC 257—400ppm. at present, the expelling wasts gas standard from automobile in our country is corresponding to the 60 centuries level of USA. some targets, such as SO₂, NO_x etc. are also higher than Japan(see table IV). therefore, in the field of environmental protection gas, we would learn the advanced experiences from foreign countries, and based on domestic situation to determine the real, possible standard for our country. At present, so many countries in the world discuss, research and develop those problems existed in the field of environmental protection.

- 1. Specialty gas department of France air liquid CO.
- (1) They adopt gravimetric method to prepare gas mixture and standard gas mixtures of air and 4N-5N N₂ which is nearly the same expelled from autômobile tail gas.

Standard content range: NO_2 in air 1-1000 ppm SO_2 in air 1-1000ppm NO in N_2 1-1000ppm relative error: 0.5%

Use 3 kind standard gas mixtures as the reference material, to determine the NO_2 , NO_2 content in automobile tail gas.

(2) Manufacture the specialized detection instruments for "environmental protection gas"

England oxygen company prepared the Asii₃/Pii₃ alarming detector, it adopted the principle of high temp., light fon detection to determine the content of Asii and Pii₃. the sensitivity is 20pph, the alarming comcentration is:Asii₃ 50ppb, Pii₃ 300ppb.

which is the same of America expelling standard. that instrument used in the production site of specialty gases.

2. Linde CO. of western Germany

Linde CO. adopted dynamic blending method to prepare the standard gas mixture of automobile tail gas. the background is 6N pure gs, its content standard is: CO in N₂ 5Oppm, CO₂ in N₂ 5Oppm, CH₄ in N₂ is 5Oppm, SO₂ in N₂ 5Oppm. relative error for blending is 0.5%, in order to gurantee the stability of standard gases, it used many kind instruments to make detection comparison and calbrated it in definite period of time.

3. AP CO. fo England

They forecasted the development of electronic gas, they considered that there would have a rapid development stage including environmental protection. they just explored how to keep stability of SO₂ in N₂. from the experimental results, in order to guarantee the SO₂ content unchanged, it must make particular treatment about gas cylinders. besides this, they research contaminant detector in particular environment. the environmental gases prepared by different countries are widely used in automobiles, aeronautics and electronic gas plant site. environmental protection gas is already the safety basis and also the important legal basis in government organizations.

4. Schedule for expelling standard of harmful gases:

Here in the following table listed the expelling standard of harmful substances in domestic and abroad for reference:

table I, Expelling standard of harmful gases in atmosphere table II, Atomospheric environmental quality standard table III, Environmental standard comparison

table IV, Standard comparison of contaminants expelled in high speed from automobilres.

Table 1 Expelling standard fo harmful gases in stmosphere

NO.	name of harmful sybstance	industries to expel harmful substances	height to expel standard gas(M)	expelling quantity	remark
1	SÍH ₄			5pmm	US expelling
2	Asil			0.05ppm	11
3	PH			0.3ppm	11
4	B.H.			0.1ppm	10
5	B ₂ H SiH ₂ Cl ₂			Sppm	111
6	NO ₂ 2 2	all sources	20	8.7mg/n	Beijing city
	2				standard
7	11	"	30	15 "	•
8	10	11	40	23 "	¦ "
9		11	60	50 "	"
10	Plurides	11	30	1.1"	
11	18	••	40	1.7 "	•
12		"	60	3.4 "	**
13	Cl	11	20	2.6 "	**
14	••	"	30	4.5 "	11
15	11	11	40	7.5 "	**
16	10.	11	60	14 "	••
17	нс1		20	1.3 "	••
18	11	••	30	2.2 "	•
19	. 11	96	40	3.7 "	
20		. 11	60	7.0 "	

Table II Atmospheirc environmental $_3$ quality standard (GB 3095-82) mg/H 3

contaminants	sampling time	lst grade	2nd grade	3rd grade
so,	day average	0.05	0.15	0.25
2			(=0.43ppm)	İ
NOx	**	0.05	0.1	0.15
×			(0.2ppm)	
CO	"	4.0	4.0	6.0
o ₃	hour average	0.12	0.16	0.20
Table II	I Environmental 3	tandard compar	ison(ppm)	
name of contaminants		SO ₂	co	NO
Japan environ	mental standard	0.64	<10	0 [×] 04

7.5

0.2

Table IV Standard comparison of contaminats expelled in high speed from automobiles

China environmental standard

(CO(%)	HC(ppm)
± 6	3000
į	(GB 3842-83)
5	2000
4	1500
3	1000
	± 6

0.43

Table V waste gas expelling standard of US automobile

Name of contaminants	CO Z	НСррт	
Expelling standard	1.5-2.3	257–400	
		'	

IV. Impression and tentative ideas

(1) Impression

Through the synthetic investigation about western Europe gas industries, we obtain an assemble impression as follows:

- 1. At present, the specialty gases which are the fundamental materials of different kind new technologies have been developed continuously in the field of product quality, new species, new applications, Productuon specialized, process automation, technology forms a complete set, products serialized. those are the main development trend of western Europe specialty gas business. those compaines established specialized specialty gas production plant. it widely used computer to do process monitoring, under the complete set technical warranty in the field of analyzing, safety, storage and transportation applications, it achieved poly-species, deep processing, serializing effectively, in regarding to the whole technology level, our country is lag behind the western Europe countries about 10-15 years.
- 2. The advanced representative technology about specialty gas business expressed in the following 6 items: i) Zero contamination, ultra-high purity, terminal purification, manufacturing technology. ii) manufacturing technology about industrial gas mixtures by means of computer iii) high automatic precise analytical detection technology by means of program control iv) gas cylinder inner wall treatment technology v) safety protection and detoxication technology about electronic gas vi) specialized instruments, meters and equipments development to form a complete set with the technology stated above. in dealing with the research work about detection and process control of dust particles and metal ions in high purity gas, those compaines are now storming the tackle key problems but it is still not putting practice in production.

3. At present, the main field of development and application of specialty gas in western Europe are electron, medical, food and environmental protection business.

Those companies put the development of new generation electronic gas in the first place, pay focus attention to the tackle key problems of ultra high purity electronic gas and high precision poly-com-ponent electronic gas mixtures. the next place in the research work is about controlling gas of environmental contamination, this reflects how the advanced countries in western Europe attach importance to the control of environmental contamination. in the field of medical diagnostic and food, they develop the applied technology and equipments forming a complete set at present.

- 4. In the field of management and development of ewstern European gas companies, they integrated the scientific research work with production, forward with near future, high technology and general technology policy, they do quick information, active management, precise technology, new products, real rpofit. During keen competition, it always kept rich forwarding ahead, therefore, it promoted the development of production and scientific research work continuously, this kind management thought is worthy for our reference and emulation.
- 5. The common points of management system in western European countries are highly trained organization, best quality, high efficiency. for instance, in the Chalon electronic gas plant of France air liquid CO.. it produced 35 kind electronic gas mixtures and one kind electronic pure gas, total employee is only 7. the large air separtation unit 100000M³/hr. of MG CO. in western Germany inustrial zone, the total employee is only 5. Crew specialty gas plant of England AP CO. is in charge of the whole England sales business, the total management person is only 8. this kind high efficiency work,

however, depends on the modernization equipment and measures, but the capable management system, well organization, person's good technical quality are also the important factors.

(II) Tentative ideas

1. Specialty gas is a kind of knowledge, it is a technology which has investment highly concentrated and synthesized its development depends on the cooporation of different departments and different specialities. in regarding the gas industries of our country. It has not strong unified management organization, and still exists "the small dispersed scale" "each does things in his own way" situation, we suggest that our government must take the unified management plan about our gas business, in those field of research work, design, production and equipment manufacture departments of industrial gas and specialty gas. organize an economic allied entity which is crossing different business and has authority.

can be integrated to tackle technical key problems and industrial production or to adopt such policies which are planned based on: district integration, key industrial foster, encourage competition, in order to facilitate the development of gas industries.

- 2. In order to shorten the difference between our specialty gas technology and foreign countries advanced level, not only depends on our self-reliance, but also imported the advanced technology and equipments from foreign countries postively. through digestion, absorbtion, and enhancement, to change the backwardness situation of our gas industries earlier.

 The France air liquid CO. has abundant technical personnel and advanced technology. its blending technology of electronic gas is completeness, pratical, well complete set equipments. it is suitable to spread in our cuntries. therefore, we suggest our plant contacting them postively. under possible conditions, make technology and trade cooperation, import key equipments, so that the electronic gas blending capacity will be formed as early as possible.
- 3. Through the real situation evaluation about western investigation, we consider the overall design about the project "Beijing specialty gas research and develop center" aided by UNIDO is reasonable, the target is positive, plan is possible. that design embodies the main development trend of advanced technology of industrialized countries. it is also compliance to our country real situation, but it can be substantiated in contents of detail activities.
 - (1) In the field of electonic gas

Based on the development foundation of electronic gas, super high purity NH3, HC1 gases, it would match with the technical import. BOP pays attention to the preparation of electronic gas mixtures, this has positive meaning for promoting development on electronic industry.

(2) In the field of medical diagnostic gas

The main force will put on the applied technical research, work about $N_2(0)$ narcotic agent, supply different kind special equipments for usees so that it can be broaden and consolidate the sales market.

(3) In the field of food gas

Mainly, we would do the research work about food preserving gas formula and applied technology of filling gas package. this task is just in begining in our country. It is a technology which has development features and heavy economic benefit.

Besides this, we would pay attention to the development and research about high purity CO_2^{\star} first of all, pay attention to applications in those fields which needs low investment, quick pay back time of profit, medium technology precision. for instance, the application of CO_2 in beer business, this will be the first project to be developed. next, we will develop the application of CO_2 in the fields of quick frozen, cigarettes, pipe freezing etc.

(4) Environmental protection gas

To prepare the poly-component gas, low concentration standard gas mixture is the key point to develop new species of environmental gases, therefore, we must grasp firmly about the construction work of precise blending room and relative research work about analytical detection. in the mean time, we should give enough attention to the complete set technology for packageing vessel.

4. Our Institute must learn the advanced experiences from foreign countries, make real modification about the present research method which is an industrial design model, it is a research method which wastes lobour, time, no economic profit and no science idea. We should pay attention to the fundamental experiments including gas-liquid equilibrium, distiller effect,

which to test the best process parameters. after the reasonable technological path has been found, then do the broaden design for pilot plant. it is strictly forbidden the non-scientific trends which are underestimate the experimental data, empiricism and workshop bad practice.

- 5. We must do the development and research work about the key technology in the field of specialty gas positively. in the 7-5 year plan period, the following technologies development must be prepared well particularly:
- (1) Development of computer soft ware for blending calculation by partial pressure method-is a revolution of blending technology, we should tackle the key points in coorperation with predominant scientific organization, academy, universities. In the mean time, integrate with the important technology, explore this technological results to give fully play about its profit for the modernization of our country as early as possible.
- (2) To strengthen the explore test of pure technology theory and its practice under possible conditions, coorperate with relative organizations to do the research work about physical characteristics of adsorbing agent, permeable membrane, package of distiller etc. modify and enrich the purity measures,
- (3) Develop the research work of package vessel technology including cylinder inner wall treatment technology and various valves, pipe fittings, instruments, regulators, flow meters, precise joints etc.
- (4) Develop the research work for detecting metal ion in high purity gas positively. in the meantime, strengthen the test explorement and follow international tracks about particles detected on technology.
- (5) Computer is the important measure for rewearch and production. fully play the computer aided function in the Tield of process monitor, analyzing tetection, scientified organization, production meanagement etc.
 - (6) Grasp firmly about the collection and collete work about toxicological

data of specialty gases, detemine the safety code of toxic gases . in the mean time, in accompaning with the technology import, digest and absorb the safety protection technology and detoxicate technology of electronic gases. put the research and production on the safety reliable foundation.

- 6. In the period of 7-5 year plan, strengthen the intention that scientific research must face to production, implement the policy "pay attention to the connotation, develop the extention". In accompaning to tackle the key problems of important product and main technology, it must intergrate with the preactical production, to arrange a batch items" short, quick, straight" directed by planning, procedures, stages, batches. solve one or two real problems for present production, so that the scientific research not only be the precurrsor of production, but also be the important guarantee condition for development of production continuously.
- 7. Enhance the quality of technical personnel is a pressing matter in the moment of our Institu te. we consider that, besides to despatch technical personnel to go abroad learning by batch and period, and to invite foreign experts for direction the work. we must adopt several methods including releasing work for studying, organizing training class, to be trained in university etc. grasp firmly the training in totation for present technical personnel.

8. We must implement the central government's strategic policy
"Economic construction must depend on the science and technology Science and
technology must face on econmoic construction". learn relative documents about
the modification of science and technology system of organization seriously.
from the standing point of our country's real situation, absorb the good
experiences of foreign countries seriously, raise the intention of "Two facing"
continuously, endeavour to make production promoting scientific research and
scientific research enhancing the production. In the field of integration
about plant and institute, we boldly explore, bravely in practice, summarize
fruitful new experiences, arouse fully the socialist enthusiasm of technical
personnel. so that they will like, keep one's mind, facilitate to work.
and early, quick, enormous to produce achievenents, devlop the new aspect of
scientific research continuously.