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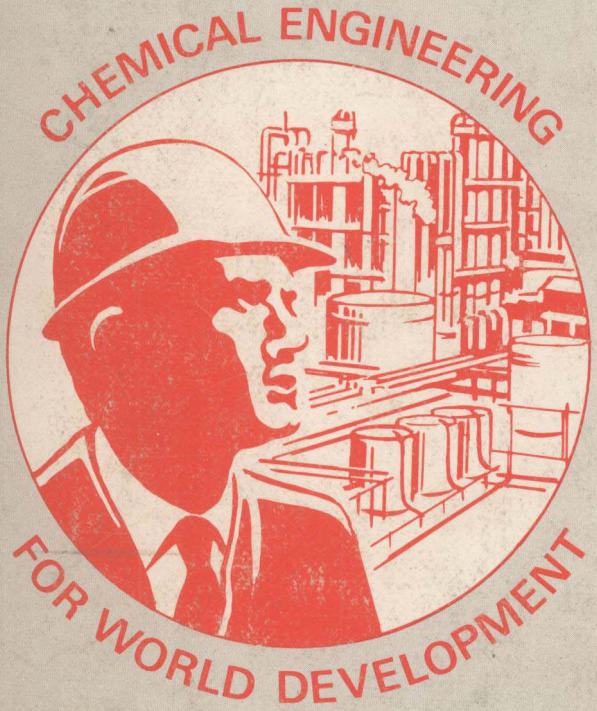

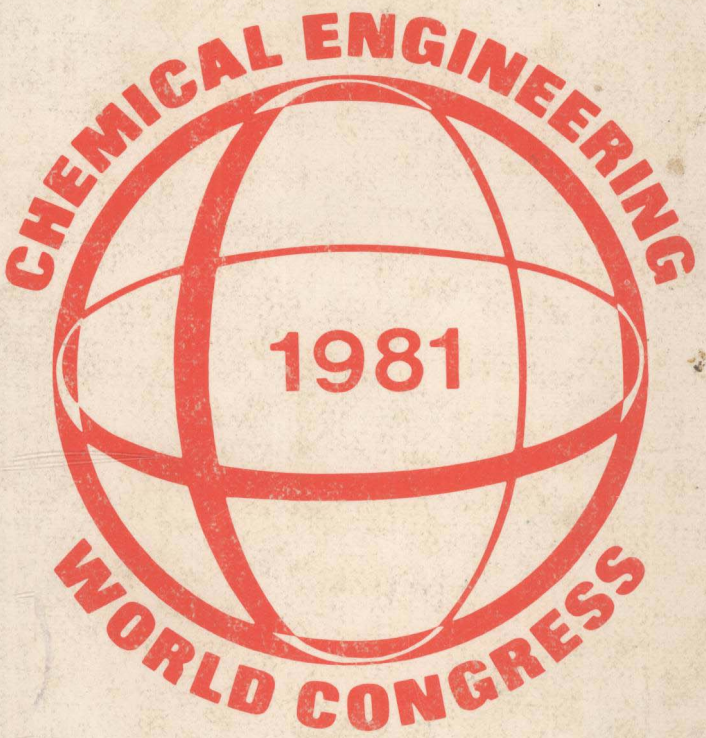

VOLUME VI

- Environmental Chemical Engineering
- Primary Industries
- Polymer Engineering

Montréal, Canada 1981 October 4-9

IX Interamerican Congress of Chemical Engineering

31st Canadian Chemical Engineering Conference

"Chemical Engineering
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VOLUME VI

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SOURCES, HEALTH EFFECTS AND CONTROL TECHNOLOGY FOR POLYNUCLEAR AROMATIC HYDROCARBONS

R E Waller

Medical Research Council, St. Bartholomew's Medical College, London.

Introduction

Polynuclear aromatic hydrocarbons are liable to be produced in the incomplete combustion of a wide range of fuels or other organic substances, and their association with the development of cancer has been recognised for more than 200 years, ever since Percivall Pott (1775) of St. Bartholomew's Hospital in London, described the unusual occurrence of scrotal cancer among chimney sweeps. This finding coupled with reports of the widespread occurrence of skin cancers among men working with coal tar led eventually to the demonstration of the carcinogenic action of extracts of soot from coal fires by Passey (1922) and to the first demonstration of the production of cancer by individual chemical substances, together with the isolation of a potent carcinogen from coal tar (Cook et al., 1932, 1933). The "control technology" required to reduce the incidence of chimney sweeps' cancer was merely the substitution of brushes and rods in place of small boys to clean the more numerous but generally smaller chimneys that emerged during the nineteenth century, while improved hygiene helped to curtail the incidence of skin cancer as a whole related to soot or coal tar.

What was not realised straight away was that while much tarry material containing carcinogenic components was deposited on the sides of chimneys, other similar material was being dispersed into the air outside, and when the use of coal for domestic heating, cooking and laundering purposes reached its peak in Britain, towards the end of the nineteenth century and up to the time of the First World War, concentrations of tarry smoke often became extremely high, leading to the "London Particular" pea-soup fogs, and to the near-permanent pall of smoke over most large cities.

It was in the inter-war years, when the experimental and analytical work of Kennaway's team was in progress, that a rising tide of lung cancer was first noticed, and the marked excess in mortality from this disease in urban as compared with rural areas suggested that air pollutants might be implicated. Stocks (1947) found an inverse relationship between lung cancer mortality and hours of sunshine as recorded in a number of large towns in England and Wales, and he concluded that either sunshine was protective or that the smoke that limited the penetration of the sun had some adverse effect itself. It was this observation that led to investigations of air pollutants as factors in the development of lung cancer, and at the same time parallel studies were set up on the role of smoking. As is now abundantly clear from the many epidemiological studies done during the past 30 years, it is the latter that is of paramount importance, and today the effects of carcinogens in urban air must be considered to be only of marginal significance.

From the beginning, the proposition that the rapid rise in lung cancer mortality might be associated with changes in air pollution seemed dubious, since concentrations of coal smoke in the major towns in Britain had remained steady or shown signs of decreasing in the inter-war years as electricity and gas displaced coal for some domestic purposes. Nonetheless, the topic of air pollution and lung cancer has been actively pursued up to the present day, and some of our own contributions in this field are outlined below.

Benzo(a)pyrene in urban air

Throughout this report, benzo(a)pyrene is used as an indicator of potentially carcinogenic components of the complex mixture of polynuclear aromatic hydrocarbons produced in the various

pyrolysis processes. Among the other compounds some have been shown in animal tests to have carcinogenic properties and many others have yielded negative results or have not been adequately tested. The relative proportions of the different hydrocarbons in a mixture can act as a valuable "finger-print" giving clues about the types of source, but that aspect is not discussed in the present paper.

As indicated above, domestic open fires fuelled with bituminous coal, in which the combustion efficiency is very low, provide a notable source of polynuclear aromatic hydrocarbons in urban air. Industrial coal-fired boilers, even when operated so as to produce black smoke, contribute little in comparison, for with their higher combustion temperatures and more adequate air supply, the emissions contain little tar.

This feature was apparent in the early measurements of benzo(a)pyrene in London (Waller, 1952), for both the concentrations in air and the proportions in the total smoke were much higher in winter, when domestic fires were in use, than in summer. The remedy has been to outlaw the use of bituminous coal for domestic purposes in major urban areas, under the provisions of the Clean Air Act of 1956. This action was stimulated by the dramatic short-term effects of pollution in the London fog of December 1952 rather than by any thought for long-term carcinogenic effects. The smoke control programme was phased over a period of about 20 years, and its progress was favoured by the emergence of natural gas as an economically acceptable alternative to coal for domestic heating purposes. While the reduction in concentrations of smoke has been very impressive, that in benzo(a)pyrene seems to have been even greater, as illustrated in Table 1. The results assembled here are not strictly comparable with one another, since the sampling sites and the analytical methods have varied over the years, but it is reasonable to conclude that concentrations are now only of the order of a tenth of what they were prior to the Clean Air Act.

Period	Sampling Site	BaP, $\mu g/1000m^3$
1949-51	County Hall	46
1953-56	St. Bartholomew's Hospital	17
1957-64	County Hall	14
1972-73	St. Bartholomew's Medical College	4

Table 1 Concentrations of benzo(a)pyrene (BaP) in air at sites in Central London, based on 24hr samples aggregated for yearly periods.

Occupational exposures

In addition to the occurrence of skin cancer among men exposed to coal-tar products, it became evident about 50 years ago that the incidence of lung cancer was also relatively high among them. Table 2 shows that nine of the "top twenty" occupations ranked in order of lung cancer mortality in the period 1921-32 were ones involving coal gas, tar or soot (Kennaway and Kennaway, 1936).

Rank	Occupation	SMR
1	Labourers, patent fuels	433
2	Gas, coke oven chargers	342
3	Gas works managers	244
5	Pavious, asphalters	209
7	Gas works foremen	197
9	Gas fitters	182
12	Chimney sweeps	170
14	Gas producer men	162
15	Gas works labourers	162

Table 2 Lung cancer mortality among occupations related to gas-works or coal tar, England and Wales, 1921-32, males. Standardised mortality ratios (SMR) in ranked order within the top twenty occupations. General population = 100.

By now the picture has changed, and in the most recent analysis

only one gas-works group features in the top twenty among a miscellany of other occupations (OPCS, 1978). Occupational risks of lung cancer have in fact become more difficult to detect, since smoking has such a powerful effect, and there are major differences in smoking habits between different occupational groups.

More detailed studies among men employed in the former coal-gas production industry have been done, making environmental measurements within the works as well as following the fates of the men individually (Doll et al., 1965, 1972, Lawther et al. 1965). An enhanced risk of lung cancer was found among those who had worked within gas-works retort houses, with heavy exposure to tarry fumes (SMR of 179), but not among men who worked elsewhere in the gas industry. It was found also that risks were greater in the older horizontal retort houses, where there were substantial escapes of tarry fumes, than in the more modern and better-controlled vertical retort houses. Measurements of the concentrations of benzo(a)pyrene in the retort house atmospheres were made in several ways, and these showed that average exposures as assessed by fixed samplers or with masks worn by the men (acting effectively as personal samplers) were of the order of 100 times those that would be experienced from urban air at the time, with peak values being far higher than that (Table 3).

Type of sample	BaP, $\mu g/m^3$
Fumes direct from retorts	2330
Above horizontal retorts	216
Long-term mean (mask samples)	3
Long-term mean (filters)	3
Urban air (annual mean, London)	0.02

Table 3 Concentrations of benzo(a)pyrene in gas-works retort houses.

The need to improve the control of tarry emissions from retorts has been circumvented in Great Britain, for the production of gas from coal has ceased almost completely, first through the introduction of oil conversion plants, but ultimately as a result of natural gas being available from the North Sea. Coke ovens remain in use: the problems of emissions of tarry fumes are analogous with those from horizontal retorts, though being in the open air risks of exposure of the workers may be somewhat less. Several studies have however demonstrated that the incidence of lung cancer among coke oven workers in steel works is above average.

Motor vehicles

Polynuclear aromatic hydrocarbons are emitted from the exhausts of both petrol and diesel engines, but the amounts are small in comparison with the (former) contributions from coal fires. There has been concern that such emissions may contribute to the incidence of lung cancer, but even among groups occupationally exposed to exhaust products there have been no clear indications to date of enhanced risks. In studies of London Transport workers extending back over the past 30 years the incidence of lung cancer among men in bus garages has not been appreciably different from that in other groups not especially exposed to diesel fumes (Waller, 1979). Correspondingly, the concentrations of benzo(a)pyrene in the garages have not been found to be particularly high even though there is much black smoke. In measurements made in the 1950's it was in fact difficult to detect the contributions of buses to the polynuclear aromatic hydrocarbon content of the garage air against the background from coal fire sources (Commins et al., 1957). Now, with this background source removed, emissions from the buses stand out more clearly, but they are still of small magnitude.

General conclusions

Polynuclear aromatic hydrocarbons are indicators of poor combustion. Where they arise inevitably in processes such as the production of coke or solid smokeless fuels, it is necessary to minimize escapes of fumes that might be inhaled by workers in the vicinity, or be dispersed in the general air. Otherwise control for the more widespread combustion sources is primarily a matter for ensuring that hydrocarbon fuels are burned completely, and in the United Kingdom by far the most effective action has been the elimination of the open coal fire in major urban areas, it being costly, if not impracticable, to produce and operate small coal-burning appliances that avoid emissions of smoke and tarry fumes. There can be little doubt that exposure to extremely high concentrations of polynuclear aromatic hydrocarbons in some occupations enhances the incidence of lung cancer, but even then the effect is not great in comparison with that of smoking, and the risks of present-day concentrations of these compounds in the general air must be regarded as small.

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THE RELEASE OF TCDD AT SEVESO

SIR FREDERICK WARNER
and
DR. A J HOWARD
Cremer and Warner Ltd
London

INTRODUCTION

On 10th July 1976 a bursting disc on a reactor in use for the production of 2, 4, 5 trichlorophenol at the Icmesa chemical plant at Seveso owned by Givaudin, a subsidiary of Hoffman La Roche and some 20 kilometres to the North of Milan, ruptured, releasing to the atmosphere at sonic velocity a chemical cocktail, containing primarily sodium trichlorophenate, ethylene glycol, and caustic soda, and in addition as an unwanted and undesirable by-product the highly toxic 2, 3, 7, 8 tetrachlorodibenzo-para-dioxin or TCDD as it is more conveniently called.

The cloud resulting from the discharge took the form of an inverted cone which drifted downwind in a S.S.E. direction. Much of the cloud carried upwards by thermal currents and by good fortune it first drifted over open fields before reaching any built up areas and centres of population. Nevertheless, deposition from the cloud occurred and both fields and buildings were contaminated.

Those are the bare facts of an incident which was classified as a national disaster, leading to the evacuation of some hundreds of the inhabitants of the affected area, the contamination in varying degrees of some 378 hectares of land and buildings and the mounting of a vast decontamination programme. The problems posed to the authorities were unprecedented as although there had been previous accidents involving the release of TCDD these had been confined to enclosed premises and none had resulted in a discharge to the atmosphere and contamination of the surrounding countryside.

The major questions facing the authorities at this time included:-

1. What was released from the reactor? How much TCDD was associated with the release?
2. What was the distribution of TCDD at ground level?
3. Would the contamination spread to water supplies or be carried outside the area of its original location?
4. What was an acceptable level of TCDD before normal activities in the area could be resumed?
5. What methods were available for decontamination?

Before referring to the problems facing the local authorities, I feel I should say a little about the mode of formation and properties of TCDD as these had a profound effect on tenable solutions to some of the problems.

TCDD is produced as a by-product during the production of 2, 4, 5 trichlorophenol which at the Icmesa factory involved the alkaline hydrolysis of 1, 2, 4, 5 tetrachlorobenzene in the presence of

ethylene glycol and sodium hydroxide at atmospheric pressure. Following the completion of the hydrolysis, the solvent, in this case, ethylene glycol, is removed by distillation at reduced pressure. If the temperature of the hydrolysate rises above about 180°C., an exothermic reaction takes place at about 230°C and the temperature rapidly rises to about 410°C with an accompanying increase in pressure and the formation of TCDD by the combination of two molecules of sodium trichlorophenate.

TCDD is virtually insoluble in water, only slightly soluble in fats, more soluble in hydrocarbons and most soluble in chlorinated organic solvents.

It is almost immobile in soil and tends to remain close to the point of application. If sprayed on crops it does not tend to be translocated. It is not readily degraded by micro-organisms and the half life appears to vary between 190-330 days.

Photolysis is negligible in aqueous suspensions or in wet or dry soil, but in methanol solution TCDD is decomposed after 24 hours exposure to ultra-violet or sunlight.

TCDD is not completely decomposed by heat until a temperature of 800°C is reached.

Evidence of the toxicity, teratogenic, carcinogenic and other effects of TCDD is largely based on animal experiments.

Although there was a considerable number of animal mortalities in the Seveso area attributable to the ingestion of contaminated herbage there have been no reported deaths among humans attributed to TCDD at least within two years of the incident.

A report prepared by the Institute Superiore di Sanita in Rome summarised the results of a two years epidemiological survey. There was increased frequency (about 0.6% in a screening of over 32,000 people) of chloracne mainly in children and young people and the condition of those affected was improving. There was some evidence of subclinical neurological damage and cases of clinically detectable polyneuropathy in adults based on the screening of 700 persons including 446 from the most heavily contaminated area. The pathological signs were most frequently found in the peripheral nervous system. There was no evidence for a positive correlation between the incidence of neurological symptoms and chloracne.

A limited percentage of cases of liver enlargement had been reported, without giving criteria for the diagnosis.

There were no abnormal foetuses in cases of therapeutic or spontaneous abortions in spite of the known teratogenic effects of TCDD on experimental animals. Thus many of the worst fears voiced by experts have not been realised at least during the first two years of clinical surveillance of TCDD exposed persons. It is however, proposed to continue surveillance for some years yet.

PRODUCTION PROCESS

The reason for the apparent occurrence of an exothermic reaction on 10th July is still not clear. According to the Final Report of the Italian Parliamentary Commission of Inquiry into the Seveso Disaster (the FRIPC Report) the process took place in four stages as follows:-

1. Reaction of a mixture consisting of 1, 2, 4, 5 tetrachlorobenzene: NaOH : Ethylene Glycol in 1:3:5.5 molar proportions plus an unspecified amount of xylene and a maximum temperature of 185°C.

2. Distillation of solvents under reduced pressure.

3. Acidulation with hydrochloric acid.

4. Recovery and purification of 2, 4, 5 trichlorophenol.

The batch quantity was about 6000 kilograms and the process was timed to allow the production of one complete batch in a 24 hour cycle involving 3 shifts.

In the first stage the reactor was heated by steam at 12 atmospheres from the factory steam system.

The evidence is that at 05.00 hours on 10th July 1976, when distillation of solvents was proceeding the production cycle was interrupted. The reactor agitator was stopped and the reaction mixture was left to cool down naturally. Being a Saturday there was no shift due to take over at this time.

Some time later in the morning the temperature instead of falling began to rise again.

There is no evidence cited in the FRIPC report that the normal maximum reaction temperature of 185°C for the first stage was exceeded, and apparently the reactor contents had cooled naturally to a somewhat lower temperature. The problem of what caused the rise in temperature to the exotermic zone remains. Efforts to reproduce this in the laboratory were reported as unsuccessful.

At 12.37 hours, a bursting disc rated at about 3.5 atmospheres and fitted to the top of the reflux column ruptured. It appears that the function of the bursting disc was to protect the equipment in the event of a faulty control of air pressure used to transfer the sodium trichlorophenate from the reactor to another vessel for acidulation. This accounts for the bursting disc being vented to the outside atmosphere through a short stack.

Press reports refer to the discharge lasting about 2 or 3 minutes and a cloud ascending to a height variously estimated between 20 and 50 metres. The immediate problem confronting the local authorities when the presence of TCDD was confirmed on 14 July 1976, was to delineate the contaminated area and to take steps to safeguard the population from further contamination.

By the evening of the next day dermatological symptoms were noted especially among some children, there were also animal mortalities. During the next few days more cases were reported including four children with severe chemical burns.

ANALYTICAL PROCEDURES AND RESULTS

Evidence of the distribution of the contamination began to accumulate from the examination of leaves, grass, soil and wipe samples from solid surfaces.

The first definition of the most heavily contaminated area was made on 24 July 1976 on the basis of analytical results and animal mortalities. By 26th July 1976 225 people had been evacuated from the most heavily contaminated area. As more results accumulated the zone was gradually extended and by the end of August Zone A as it was called, covered 108 hectares and the number of people evacuated totalled over 730.

Further analytical results from areas at greater distance from the point of discharge showed lower concentrations of TCDD and this led to the first definition of Zone B. In this area evacuation was not proposed, but children under the age of 12 were removed during daylight hours, as were women in the first three months of pregnancy. There were also rigorous controls on food and water supplies. Zone B comprised of some 269.4 hectares in all.

In addition the authorities defined a Zone of "respect", Zone R, a buffer zone between areas contaminated and areas considered to be unaffected by TCDD. Zone R consisted of some 1430 hectares.

Obviously, this definition of contaminated areas remained somewhat tentative until further samples had been analysed. Fortunately, as the result of earlier industrial accidents, realisation that herbicides such as 2, 4, 5 trichlorophenoxyacetic acid might contain significant traces of TCDD and the massive use of these herbicides as defoliants in the Vietnam War, analytical procedures for the determination of very small quantities of TCDD already existed. By using gas chromatography and capillary columns combined with mass spectrometry it had been found possible, under the most favourable conditions to determine as little as 5 picograms of TCDD.

The first results obtained from soil, vegetation, and wipe samples produced highly variable results. However, by mobilising the resources of laboratories in Milan and introducing trained personnel and a rigid sampling routine more consistent and reliable results were obtained.

These results established three important factors. Firstly, the high values found on vegetation particularly trees indicated that a substantial part of the contamination had been deposited on upstanding material. The second factor was the low incidence and level of TCDD contamination found inside buildings. This was accounted for by the fact that the discharge occurred on a Saturday when most factories, workshops, schools and other public buildings were closed. In addition as was customary because of the heat,

most houses had almost all their windows shuttered. Thirdly, there was no evidence of contamination of well water and other drinking water supplies either with TCDD or the more water soluble 2, 4, 5, TCP sodium salt. Apparently there was little penetration of the contamination in the soil.

Soil samples were obtained by removing the surface vegetation and taking cores 6.5 cm in diameter and 7 cm deep. Three cores were collected at each sampling point, two were combined for TCDD determination and the third retained as a reference sample. The results were expressed in micrograms per square metre.

The main problem was the clean up of the samples before presentation to the gas chromatrograph and this involved solvent extraction and column chromatography.

The efficiency of recovery of TCDD was checked by spiking uncontaminated soil with known amounts of TCDD. It was found that when TCDD levels were 0.01 ug / 500 g of soil or more recovery averaged 86%. When the soil concentration was as low as 0.005 µg / 500 g the recovery dropped to 66%. All results were corrected for these recovery values the lower factor being applied when the total TCDD content did not exceed 9 µg.

Within six months of the accident the number of samples of all kinds collected and analysed for TCDD by the GC - MS method amounted to several thousands and it reflects great credit on all concerned that such high standards were maintained.

LEVEL OF CONTAMINATION AND ACCEPTABLE LIMITS

It was about this time that Cremer and Warner were retained as consultants by the Lombardy regional authority to advise on decontamination procedures.

At that time the exact quantity of TCDD emitted was still in doubt as was the area over which significant quantities had been deposited. It was therefore decided to attempt to model the incident as a check on the information then available and hopefully to provide additional information on the dispersion of TCDD.

In general the model confirmed most of the assumptions made about the incident and gave reassurance that high levels of contamination would not be expected further afield. There were however, very high levels of TCDD measured close to the source (up to 20,000 $\mu g/m^2$) which, were not reflected in the model calculations. This would be explained by the emission of larger droplets of particles as the exit velocity subsided towards the end of the discharge. The general agreement over the rest of the area tended to confirm that a total emission of about 2 kg of TCDD was of the right order. For further details of the modelling technique I would refer you to a paper presented to the Royal Meteorological Society by my colleague, P J Comer.

One of the most difficult problems facing the authorities was the definition of an acceptable level of contamination so that de-

contamination procedures could be reviewed and developed in the light of this level.

Although there was a considerable volume of data on the toxicity of TCDD this was mainly based on animal experiments. Clearly it was an extremely toxic material the LD_{50} for male guinea pigs being about 5 µg/kg.

There was also a considerable amount of information arising from previous industrial accidents with TCDD on the effects on humans, but unfortunately most of this was unquantitative.

The Medico-Epidemiological Commission examining this problem ultimately concluded that a tolerance limit of 5 $\mu g/m^2$ for the soil provided a very large safety margin.

Quite independently the consultants arrived at a similar figure when asked to consider criteria for redefining the boundaries of Zone B.

For the interior of buildings where contact was more likely to be prolonged the Commission recommended a value of 0.1 ug/m^2 and for ground in the immediate vicinity of schools, public buildings and work-places a limit of 0.75 $\mu g/m^2$ was adopted.

The adoption of these tolerance limits made possible the development of a plan for the systematic decontamination and rehabilitation of Zones A and B.

DECONTAMINATION

At the time of the accident the weather was hot and dry and most of the fields were covered with thick vegetative growth standing ½ to 1 metre tall. From the first analytical results it seemed likely that the heaviest contamination remained deposited on the vegetation. It was suggested by Givaudin that spraying with soil stabilizing resin emulsions might seal the contamination to the vegetation and bare earth and that the contamination could be removed by cutting and collecting the vegetation.

Unfortunately permission to proceed was not immediately obtainable and the dry weather ended abruptly in a series of violent thunder-storms and heavy rain. The contamination was thereby transferred from the vegetation to the soil and subsequent decontamination rendered more difficult.

Of the decontamination procedures available incineration, photolysis and microbiological degradation were all given serious consideration.

After a study of the conditions required for destruction of TCDD by incineration the consultants Cremer and Warner concluded that it was perfectly possible to design a suitable incinerator and therefore recommended the installation of such an incinerator for the de-struction of contaminated combustible materials such as vegetation, rabbit hutches, chicken houses, sheds, etc., and materials used for decontamination such as swabs, vacuum cleaner contents, protective clothing, etc.

In the event, however, local feelings against an incinerator reached such a pitch that it became politically impossible to adopt this method.

Photolysis required the addition of a solvent and a hydrogen donor and Givaudin suggested and tried in a field experiment spraying of herbage with a mixture of olive oil and cyclohexane. However, in spite of positive indications of TCDD reduction the method was not adopted as the arrival of autumn produced with it a period of heavy rain.

Although a review of the literature indicated the possibility of TCDD degradation by soil micro-organisms, the lengthy time involved to prove this method precluded it from inclusion in the decontamination plan for residential areas.

Thus the general concept of decontamination became limited to physical removal of contaminated materials and vegetation, from the lesser to the more contaminated areas and the stockpiling of these until a de-cision could be made for their ultimate disposal. The interiors of buildings and houses were decontaminated by a combination of vacuum cleaning and washing down with detergents.

All operations within the boundary of Zone A had to be carried out under the strictest control of access and procedures similar to those used in handling radioactive materials were adopted.

A main stockpile for contaminated materials was set up in Zone A 5 and materials were stored under conditions which would prevent re-dispersion and public access. It was also necessary to prevent the spread of contamination by the movements of rats and other animals.

The immediate objectives of the decontamination programme as approved by the Technico-Scientific Commission in Rome, were (a) to dispose of any "high spots" of TCDD contamination in Zone B, and (b) to complete the decontamination of the residential area in Zones A 6 and A 7 in order to permit the displaced occupants to return to their homes as soon as possible.

It was decided that in open fields a TCDD level of less than 15 $\mu g/m^2$ would not be disturbed, but that access would be restricted by fenc-ing. Above that level the top 10 cm would be scraped off and removed to the stockpile in Zone A 5. In private gardens the topsoil would be removed to a depth of 10 cm wherever the surface concentration exceeded 5 $\mu g/m^2$.

Vegetation from gardens was collected in plastic sacks and further enclosed in plastic silos at the long term storage site.

The most highly contaminated zones were closed to all unauthorized access by the erection of a tall fence of corrugated GRP sheets.

Decontamination of the exteriors of properties was carried out with a combination of high intensity vacuum cleaning and high pressure water jets.

All decontamination work was checked by GC - MS of samples.