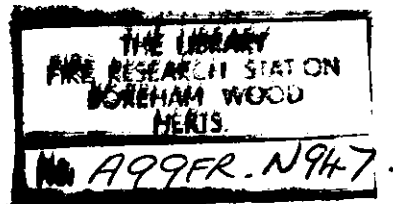# Fire Research Note
No. 947

THE PRODUCTION OF FREE TOLYLENE DIISOCYANATE
FROM THE THERMAL AND THERMAL-OXIDATIVE
DECOMPOSITION OF FLEXIBLE POLYURETHANE FOAMS

by

W. D. WOOLLEY and ANN I. WADLEY

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THE PRODUCTION OF FREE TOLYLENE DIISOCYANATE
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SUMMARY

The production of free TDI from the thermal and thermal oxidative decomposition of polyester and polyether flexible polyurethane foams has been monitored at temperatures between 200 and 800°C. The decompositions were undertaken in an all-glass system and the TDI collected in dry toluene and analysed by gas chromatography and mass spectrometry. The polyester foam releases a maximum of 1.1 and 1.15 weight per cent of TDI in nitrogen and air atmospheres respectively whereas the polyether foam generates respectively 0.9 and 0.7 weight per cent. These maximum yields are recorded at the relatively low temperatures of 250 to 300°C. At temperatures above about 800°C in nitrogen and 700°C in air, TDI is completely destroyed in the decomposition apparatus.

Realistic fire tests involving TDI based flexible foams are required in order to evaluate the hazard of free TDI in real fire situations. Additional information on the toxic effects of TDI may be required for this appraisal.

KEY WORDS: Gas chromatography, mass spectrometry, polyurethane foam, pyrolysis, Tolylene diisocyanate, toxic gas.

DEPARTMENT OF THE ENVIRONMENT AND FIRE OFFICES' COMMITTEE

JOINT FIRE RESEARCH ORGANIZATION

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1. INTRODUCTION

Recent work in this laboratory has shown that the thermal decomposition
of flexible polyester and polyether polyurethane foam involves a depolymerisation
process[1,2]. Although it has been established that the tolylene diisocyanate
(TDI) part of each foam is released mainly as a polymeric material (termed
yellow smoke) this does not preclude the possibility of the release of some
free TDI.

TDI is known to be a highly toxic material[3,4] and currently the T.L.V.*
is 0.02 p.p.m. This value is a ceiling level. At 0.5 p.p.m. the material
is a severe irritant and exposures to concentrations reported as 'very low'
can cause asthmatic-like conditions with sensitization to further TDI inhalation.
Although it is usual for anyone exposed to these concentrations to return to
normal health after they are removed from the TDI atmospheres, there is
evidence that repeated attacks from TDI exposure may give permanent injury.
The atmospheric concentration directly hazardous in a short (ie 10 minute)
exposure is unknown.

The release of free TDI from the thermal decomposition of polyurethane
foams could, in principle, present a serious toxic risk to persons involved in
fire situations either by accident or in the course of fire fighting.

The work of this report was undertaken to determine the quantities of
free TDI released during the thermal and thermal oxidative decomposition of
polyester and polyether flexible foams. This work has also involved
decompositions of the yellow smokes from the foams in order to gain information
of the source of the TDI.

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* sometimes termed M.A.C.

Although TDI is a relatively stable liquid (B. Pt of 2:4 isomer is 251°C) for bulk handling in the manufacture of polyurethane foams, it is a difficult material to handle and analyse particularly at low concentrations in vapour or solution form. It is very reactive and combines readily with materials with active hydrogen atoms (eg amines, alcohols, water etc). In vapour form it is sensitive to certain surfaces, particularly metals, by polymerisation.

In this report careful attention will be directed towards the experimental systems and procedures which will be given in detail. Basically these consist of:-

(i) Development of a chromatographic procedure for the analysis of TDI in dilute solution in toluene.

(ii) Evaluation of the observed gas chromatographic peak by direct seeding techniques and mass spectrometry to confirm that the peak is TDI.

(iii) Development of an all-glass decomposition system and a collection method for TDI produced from the thermal decomposition of the foams.

(iv) Evaluation of the decomposition system by introducing known quantities of TDI and monitoring the TDI recovered at various furnace temperatures.

(v) Evaluation of the 'TDI' peak (by direct seeding techniques and mass spectrometry) obtained during the thermal decomposition of the foams to confirm that the peak is TDI.

It will be noted in the report that all mass spectrometric experiments involving TDI were carried out using a direct insertion probe because of the complete destruction of TDI in the interface equipment coupling the mass spectrometry to the gas chromatograph[5].

In the report the quantities of free TDI released during the thermal decomposition of the polyurethane foams and yellow smoke at temperatures between 200 and 800°C are determined. Although originally designed for inert atmospheres only it was found that the production of free TDI could also be monitored in air atmospheres. The presence of the moisture from the thermal-oxidative decomposition did not apparently destroy the TDI.

2. EXPERIMENTAL

a) The decomposition and collection system

The decomposition and collection system is shown diagrammatically in Fig. 1. This consists of a quartz-glass decomposition tube (20 mm o.d.) fitted into a tube furnace (maximum temperature $1000^{\circ}C$) as shown. The exit end of the decomposition tube is tapered to 10 mm o.d. and heated to $150^{\circ}C$ with electrical heating tape. A right-angled borosilicate collection tube (6 mm o.d.) is fitted into the main decomposition tube as shown and a gas-tight seal obtained with the silicone rubber tubing. It is important that the collection tube penetrates into the hot zone produced by the electrical heating tape.

Weighed samples (10 mg) of polyurethane foam or an equivalent weight (2.5 mg) of yellow smoke are placed into a small ceramic boat and injected into the furnace using an external magnet on the steel plug. Dry air (B.O.C. medical grade) or oxygen-free nitrogen (B.O.C. white spot grade) at 50 ml/min provides the decomposition atmosphere and carries the TDI to the collection tube where the carrier gas is bubbled through approximately 1 ml of dry toluene for the extraction.

After a decomposition period (15 minutes) the toluene and collection tube are removed, the toluene is made up to 1.0 ml (to replenish the material lost by evaporation) and samples of the toluene removed with a 'Pasteur' pipette and washed repetitively through the collection tube. This washing process is important since the TDI tends to condense on the cold parts of the collection tube before reaching the toluene; hence the importance of the collection tube penetrating the hot zone of the main decomposition tube.

When using the system care must be exercised to prevent a 'suck back' of the toluene into the furnace particularly when using air as the carrier gas. This can occur when the cold boat and sample are injected into the furnace. This operation should be carried out carefully.

b) Chromatography

Chromatographic analyses were undertaken using a Hewlett Packard 5750 research gas chromatograph equipped as a dual column instrument with flame ionization detectors. TDI was eluted from stainless steel columns (2 m x 3.2 mm) packed with 2.5% silicone gum rubber (E 301) on AW-DMCS chromosorb G (80-100 mesh) temperature programmed from $80^{\circ}C$ at $4^{\circ}C/min$ with a helium flow rate of 40 ml/min.

c)   Mass spectrometry

Mass spectra were obtained using a fast scanning version of the A.E.I.
organic MS 20.   Because of difficulties in introducing TDI eluted from the
chromatograph into the mass spectrometer using the conventional interface
systems (helium separator), spectra were obtained using a direct insertion
probe.   All spectra were recorded at 70 eV.

d)   Materials

The polyester and polyether flexible foams used in this report are
identical to those used in earlier work[1,2] and were obtained commercially.
Both foams were prepared from TDI.   The polyester foam is based on a lightly
branched polyester of adipic acid and diethylene glycol and the polyether foam
prepared from a polyether produced from glycerol, propylene oxide and ethylene
oxide.   A very small addition of a green pigment is present with the polyether
foam.

The TDI used in calibration, direct seeding and other experiments was
obtained commercially as 2:4 tolylene diisocyanate (approximately 97%).   For
use it was diluted with dry toluene as necessary.   These solutions were
discarded regularly and replaced with fresh solutions during the experimental
work of this report.

3.   RESULTS

a)   Chromatography and mass spectrometry of TDI

A number of different column systems were examined for the analysis of
TDI.   The column finally adopted was a silicone gum rubber on chromosorb G
temperature programmed as recorded in the experimental section.   A chromatogram
showing the separation of TDI from toluene by injecting 10 μl of a solution of
0.1% TDI in toluene is recorded in Fig. 2.   The TDI used in this experiment was
obtained commercially and quoted as 97% of the 2:4 isomer.   No separate peak
which could be attributed to the 2:6 isomer was found even under very high
sensitivity.   The separation of the isomers by chromatography is very difficult
and it is unlikely that this system would show any differentiation between
the two isomers.   Such a separation was not required for the work of this
report.

Experiments involving peak area integration of the toluene and TDI respectively of Fig. 2 showed that the TDI peak was consistent with a 0.1% solution, indicating little or no loss of the reactive TDI during chromatography. Attempts to introduce the TDI, as eluted from the chromatograph, into the mass spectrometer via the helium separator gave no spectrum at all for the component. A further test involving the introduction of a 5% solution of TDI in toluene directly into the helium separator gave a distinct toluene spectrum but no TDI spectrum.   It is presumed that the TDI is lost by polymerisation inside the hot inlet system of the mass spectrometer.

A small sample of the TDI solution (5%) was then injected directly into the ion source of the mass spectrometer via a direct insertion probe.   After increasing the temperature of the probe slowly to preferentially release the toluene the distinct spectrum of TDI as shown in Fig. 3 was recorded.   The spectrum shows a general hydrocarbon background (probably from impurities in the toluene) and superimposed on this, the distinct spectrum of TDI characterised particularly by ion masses 174, 173, 146, 145 and 132.   The base ion (174) is also the molecular ion.   A reference spectrum of TDI listing the 10 most abundant ions[6] with intensities (based on a maximum of 1000 units) is given in Appendix II and is in good agreement with the experimental intensities of the 174, 173, 146, 145 and 132.

A chromatographic. experiment was then carried out to collect the TDI, as eluted from the chromatograph (from the injection of 10 μl of 5% TDI in toluene), by bubbling the chromatographic carrier gas through 0.2 ml dry toluene during the elution of the TDI peak.   A sample of the collected TDI in toluene was introduced into the mass spectrometer with the direct insertion probe giving a substantial toluene spectrum plus a distinct base peak of 174 (also the molecular ion) of TDI.   Other fragments were masked by the hydrocarbon impurities of the toluene.

This chromatographic and mass spectrometric evidence clearly indicated that the chromatographic analysis of TDI in this way was valid.

b)   Collection experiments of TDI from pyrolysis equipments.

A variety of decomposition and collection systems was evaluated for studies of the production of TDI from the thermal and thermal oxidative decomposition of the polyurethane foams.   These systems were first evaluated by introducing a known quantity of TDI into the hot zone (maintained at $200^{\circ}C$) in a stream of nitrogen and analysing the TDI recovered by bubbling the gases

through 1 ml of toluene by chromatography. These preliminary tests showed that unless a number of important criteria were followed rigorously then the recovery of TDI, even on adding substantial quantities of TDI to the furnace, could easily be zero.

The decomposition system finally adopted has been discussed in detail in the experimental section and is shown in Fig. 1. It is particularly important that the entire apparatus is constructed of glass and flame-cleaned regularly. The collection tube, as stressed in the experimental section, must penetrate the hot decomposition tube to avoid TDI condensing before reaching the collection tube.

Fig. 4 shows the TDI recovered, as a percentage of the theoretical, by injecting 2.5 μl of 5% TDI in toluene into the furnace in nitrogen and air streams at temperatures between 200 and 800°C. The per cent of theoretical figure for each experiment is derived by comparing the concentrations of TDI (by chromatography) obtained firstly by introducing the 2.5 μl of 5% TDI solution into the furnace and collecting it in the 1 ml of toluene* with that obtained secondly by introducing the 2.5 μl of 5% solution directly into 1 ml of toluene.

In Fig. 4 at 200°C, 88 and 83 per cent of the theoretical are recovered in nitrogen and air atmospheres respectively. TDI can be monitored in nitrogen to 850°C and in air only to 650°C. It is not known whether the losses of TDI at the higher temperature are associated with thermal and/or thermal oxidative decomposition or polymerisation. Alternatively, the losses of TDI at high temperatures may be associated with an 'instrumental' destruction. This point will be discussed further in a later section.

c) Qualitative decomposition experiments

Decomposition experiments with 10 mg of the polyester and polyether foams were carried out in a nitrogen atmosphere at 300°C. Analysis of the 1 ml toluene solution in each case showed a distinct chromatographic peak eluted at the exact retention time of TDI. Direct seeding experiment where small quantities of TDI were added to the furnace during the decomposition experiments increased the 'TDI' peak from each foam without any doublet formation. The concentration of TDI in the toluene obtained by the direct decomposition experiments was too low to attempt any collection experiments during chromatography for mass spectrometric confirmation of the material.

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* For 15-minute periods

Instead, samples of the TDI in toluene as obtained directly from decomposition experiments involving the two foams were introduced into the mass spectrometer in turn via the direct insertion probe.   Under high mass spectrometric sensitivity the solution from each foam showed, in addition to toluene, a complex series of ion masses but ion masses at 174, 173, 146, 145 and 132 were distinct.   Further the relative ion intensities of these masses were consistent with those obtained from the 5% TDI in toluene solution.

These tests confirmed that the material being monitored during the thermal decomposition of the foams was free TDI.

d)   Quantitative decomposition experiments

Before undertaking detailed quantitative experiments, the sensitivity of the chromatograph over the working range of the TDI solution was obtained. This was effected by preparing a series of TDI solutions in toluene (1 ml) of known concentrations and analysing by chromatography to obtain the area of the TDI peak in each case.   Fig. 5 shows the TDI peak area, ie the integrator counts, in arbitrary units plotted directly against the weight (μg) of TDI in 1 ml of toluene.   When plotted in this way the calibration curve gives a direct reading, during the thermal decomposition experiments, of the quantity of TDI released.   A non-linearlity of the calibration curve is evident in Fig. 5 but the reason for this is not understood.

Decomposition experiments were undertaken with the polyester and polyether foams (10 mg) and polyester yellow smoke* (2.5 mg) in nitrogen and air atmospheres at temperatures between approximately 200 and 800°C.   The integrator counts and the yields of TDI (as obtained from the calibration graph of Fig. 5) are recorded in Appendix I.

The yields of TDI in microgrammes from 10 mg of foam (or 2.5 mg of yellow smoke) plotted as functions of temperature for the decomposition in nitrogen are recorded in Fig. 6.   The equivalent data for the decompositions in air are shown in Fig. 7.   For comparison purposes both figures are plotted on equivalent scales.   Division of the microgramme yields by a factor of 100 gives the weight per cent conversion of foam to TDI.   For convenience 1.0% weight conversion is marked on each graph.

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*The polyester yellow smoke was prepared under inert conditions.
2.5 mg is the approximate weight of smoke obtained from 10 mg of foam.

The decomposition experiments were undertaken at temperatures up to 800°C in nitrogen and 700°C in air since earlier work (See Fig. 4) had established that TDI would be destroyed above these respective temperatures. The fact that the observed yields of TDI from the thermal and thermal-oxidative decomposition of the foams decreases to virtually zero at 800°C in nitrogen and 700°C in air is further confirmatory evidence that the species under investigation from the foams is free TDI.

4. DISCUSSION

TDI recovery experiments show that at temperatures above about 400°C there is a rapid decrease in the TDI recovered from the furnace system. TDI can be monitored at temperatures up to about 850°C in nitrogen and 650°C in air. The experiments of this report do not indicate the mode of this high temperature loss. It is likely that it associated with gas phase decomposition or polymerization but could be an instrumental feature in that the glass walls of the furnace promote the loss.

In the inert atmosphere, the production of TDI is measured as a distinct maximum at 300°C for each foam. This maximum cannot be attributed to the temperature-dependent recovery efficiency since it occurs within the relatively 'flat' recovery zone between 200 and 400°C (see Fig. 4). The maximum yields (1.1 and 0.9 weight per cent for the polyester and polyether foams respectively) are therefore real but the TDI released at temperatures in excess of about 400°C may be somewhat low. No attempts will be made in this report to modify the high temperature yields of the foams by reference to the recovery data of Fig. 4. This would not be a valid exercise until the mode of decomposition has been established.

In air, the maximum yields (1.15 and 0.7 weight per cent for the polyester and polyether foams respectively) are recorded at 300°C for the polyester foam and at the slightly lower temperature of 250°C for the polyether material. The relatively low yield for the polyether foam at 250°C may be associated with the general instability of polyether foams to oxidative environments. There does not appear to be any evidence that the presence of moisture from the oxidative decomposition of the foams is seriously reducing the yields of TDI. Possibly the condensation of TDI (B.Pt 251°C) occurs on the collection tube at temperatures sufficiently high to prevent water condensation and therefore the two materials become physically separated during the collection cycle.

Again, in air, no attempts will be made to modify the high temperature yields of the foams by reference to recovery data. However, in both air and nitrogen atmospheres it must be considered that the maximum yields of TDI (at 250 to 300°C) could be 10 to 15 per cent higher than recorded because of the recovery efficiency (Fig. 4). This modification will not be incorporated into this report because the collection efficiency during actual decomposition experiments where the TDI is released slowly may not correlate exactly with that observed during the sudden injection of TDI into the furnace.

In both air and nitrogen atmospheres there is evidence that free TDI is released from the decomposition of the polyester yellow smoke. It must be noted that the preparation of yellow smoke[1] involves heating the foam to 300°C. The release of free TDI from the yellow smoke at 300°C therefore represents a second heating cycle. It is not clear from this data alone whether the free TDI released during the decomposition of the foams is produced directly or indirectly via the decomposition of the yellow smoke. Both routes may be contributory.

In fires and allied situations the most serious conditions are likely to be encountered where the foam is decomposed at a relatively low temperature and the decomposition gases released without passing through a hot zone. A hot zone above 700-800°C appears to completely destroy TDI but the mode of destruction is not known. The decomposition atmosphere has little effect on the production of free TDI with the polyester foam but oxidative environments reduce the maximum yields with the polyether material.

At 20°C and normal atmospheric pressure 1 p.p.m. of TDI is equivalent to 7.24 mg/m[3]. Thus the decomposition of 1 gram of polyester and polyether foams in a metre[3] under optimum conditions for formation of TDI could produce 1.5 and 1.2 p.p.m. in nitrogen and 1.6 and 1.0 p.p.m. in air respectively. These concentrations are of serious irritancy level and well above the TLV ceiling level of 0.02 p.p.m.[3,4].

The level likely to be immediately hazardous in fires is not known. Saturated air[3] at 20°C is quoted as containing approximately 13 p.p.m. although there is some doubt about this figure. Based on a saturation figure of 13 p.p.m. it is unlikely that concentration of TDI hazardous to life within minutes would be attained during the normal use of TDI at room temperature. An exposure of 11 p.p.m. for 4 hours is reported to have killed 50% of rabbits within 14 days[3].

In fire situations a ceiling level of 13 p.p.m. due to air saturation at room temperature is not valid. Firstly atmospheres in fires can be tolerated at temperatures far in excess of normal room temperature and secondly TDI if migrating from a very hot environment and then cooling to a breathable temperature can be inhaled as an aerosol or condensed on solid matter or as vapour.

It is a recommendation of this report that realistic fire tests should be undertaken involving TDI based foams to monitor the concentration of TDI in the fire gases. This work must involve satisfactory sampling, collection and analytical methods and should be evaluated as a complete system because of the real possibility of TDI destruction in the sampling lines. Additional information relating to the toxicity of TDI, particularly the atmospheric concentration hazardous to life within a short exposure may be required for this assessment.

5. CONCLUSIONS

(1) Free tolylene diisocyanate (TDI) is released during the thermal and thermal oxidative decomposition of flexible polyester and polyether polyurethane foams.

(2) In inert atmospheres the maximum yields of TDI (1.1 and 0.9 weight per cent for the polyester and polyether foams respectively) are recorded at about 300°C.

(3) In air the maximum yield (1.15 weight per cent) of TDI is observed at 300°C for the polyester foam whereas the maximum yield (0.7 weight per cent) for the polyether foams occurs at about 250°C.

(4) High temperatures tend to destroy TDI. Free TDI injected into the furnace is observed only at temperatures up to 800°C in inert atmospheres and 700°C in air.

(5) The decomposition of 1 gram of polyester or polyether foam in 1 m³ of air would under the most favourable conditions for the production of TDI, give 1.6 and 1.0 p.p.m. respectively.

(6) Realistic fire tests involving TDI based flexible polyurethane foams are required in order to evaluate the concentration of TDI in real fire situations. Further information on the toxic effects of TDI may be required to assist the overall appraisal.

6.   REFERENCES

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7. APPENDIX I

Integrator counts and yields of TDI from the thermal decomposition of polyester and polyether foams in nitrogen

Polyester foam (10mg)

Temperature (°C)	Integrator counts (arbitrary units)	TDI μg per 10 mg foam
200	1000 (0)*	0
250	2442 (3)	24
275	6374 (3)	54
287	9701 (3)	77
300	1060 (4)	83
312	1455 (4)	109
325	8347 (3)	67
350	1635 (3)	17
400	2177 (3)	22
500	2729 (3)	26
600	2113 (3)	21
700	2492 (3)	24
800	5000 (2)	5

Polyether foam (10mg)

Temperature (°C)	Integrator counts (arbitrary units)	TDI μg per 10 mg foam
200	2964 (2)	3
250	7121 (3)	59
275	1190 (4)	92
300	1067 (4)	84
325	1191 (4)	92
350	7441 (3)	61
400	3615 (3)	33
500	2832 (3)	27
600	2964 (3)	28
700	2689 (2)	3
800	0	0

*Number in brackets denotes powers of 10; for example 2442 (3) = 2442000 counts.

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APPENDIX I (cont'd)

Integrator counts and yields of TDI from the thermal decomposition of polyester and polyether foams in air

Polyester foam (10mg)

Polyether foam (10mg)

Temperature (°C)	Integrator counts (arbitrary units)	TDI µg per 10 mg foam
250	3017 (3)*	28
275	8696 (3)	70
287	1529 (4)	114
300	1239 (4)	95
312	1091 (4)	85
325	9668 (3)	77
350	6886 (3)	57
500	4116 (3)	37
600	8159 (2)	9
700	0	0

Temperature (°C)	Integrator counts (arbitrary units)	TDI µg per 10 mg foam
200	0	0
225	3098 (3)	29
237	8853 (3)	71
262	8222 (3)	66
300	4717 (3)	41
400	2182 (3)	22
500	8378 (2)	9
600	4620 (2)	5
700	0	0

*Number in brackets denotes powers of 10; for example 3017 (3) = 3017000 counts.

APPENDIX I (cont'd)

<u>Integrator counts and yields of TDI during the thermal decomposition of</u>
<u>yellow smoke (polyester) in nitrogen and air</u>

Yellow smoke (2.5 mg) in nitrogen   Yellow smoke (2.5 mg) in air

Temperature ($^\circ$C)	Integrator counts (arbitrary units)	TDI μg per 10 mg foam[ø]
250	4960 (2)[*]	5
275	8376 (2)	9
300	1437 (3)	15
325	2366 (2)	3
400	5000 (2)	5
600	1021 (3)	11
700	1757 (2)	2

Temperature ($^\circ$C)	Integrator counts (arbitrary units)	TDI μg per 10 mg foam[ø]
200	0	0
250	4720 (2)	5
300	3087 (2)	3
400	5849 (2)	6
500	1578 (3)	16
600	1741 (3)	18
700	0	0

[*]Number in bracket denotes powers of 10;   for esample 4960 (2) = 496000 counts

[ø]The content of yellow smoke of 10 mg foam is approximately 2.5 mg.

8. APPENDIX II

Mass spectra of TDI

(1) Reference spectra from Cornu and Massot[6]

Ion mass	174	145	28	27	173	51	39	146	44	132
Intensity	1000	404	385	276	272	240	220	216	199	183

(2) Certain ion masses and intensities from
     experimental spectrum*

Ion mass	174	145	146	173	132
Intensity	1000	393	380	209	160

*Ion masses at 28, 27, 51, 39 and 44 are masked by
the hydrocarbon background.

Borosilicate
collection tube

Silicone
rubber tubing

Electrical
heating tape

Quartz
decomposition tube

Dry air or nitrogen

Furnace

1ml toluene

Ceramic boat
and sample

Steel plug
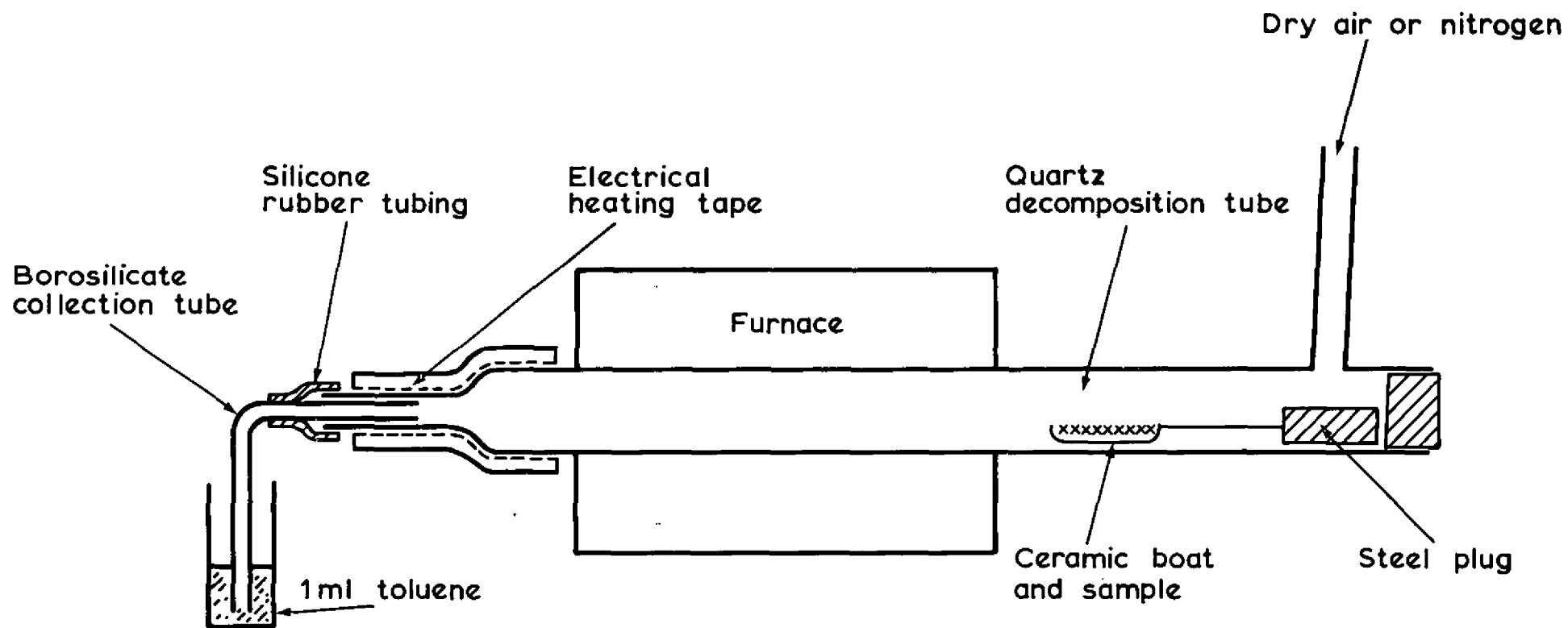
FIG. 1   THE DECOMPOSITION AND COLLECTION SYSTEM FOR TDI STUDIES
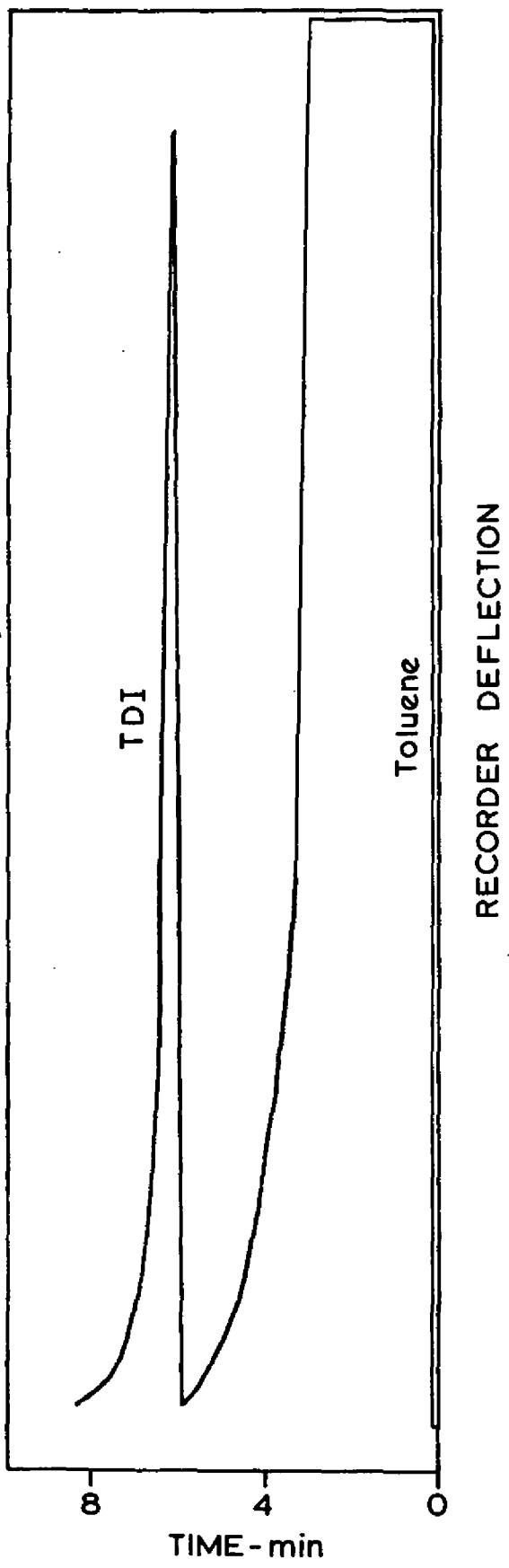
FIG. 2 CHROMATOGRAM OF TDI AND TOLUENE
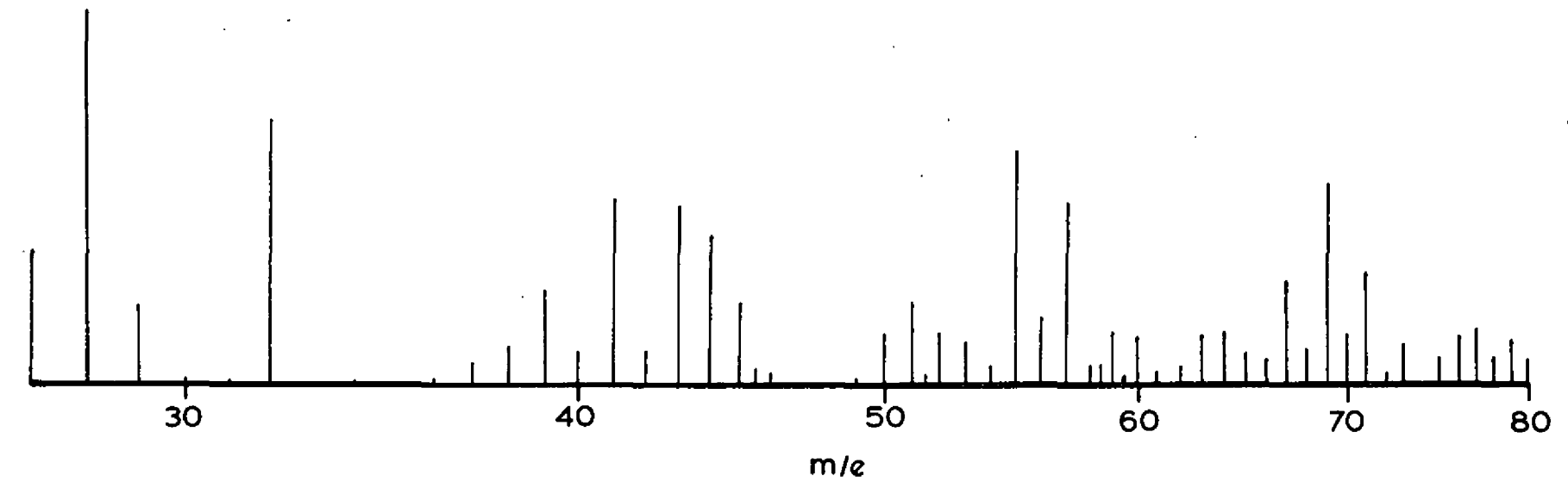USING A SILICONE GUM RUBBER COLUMN
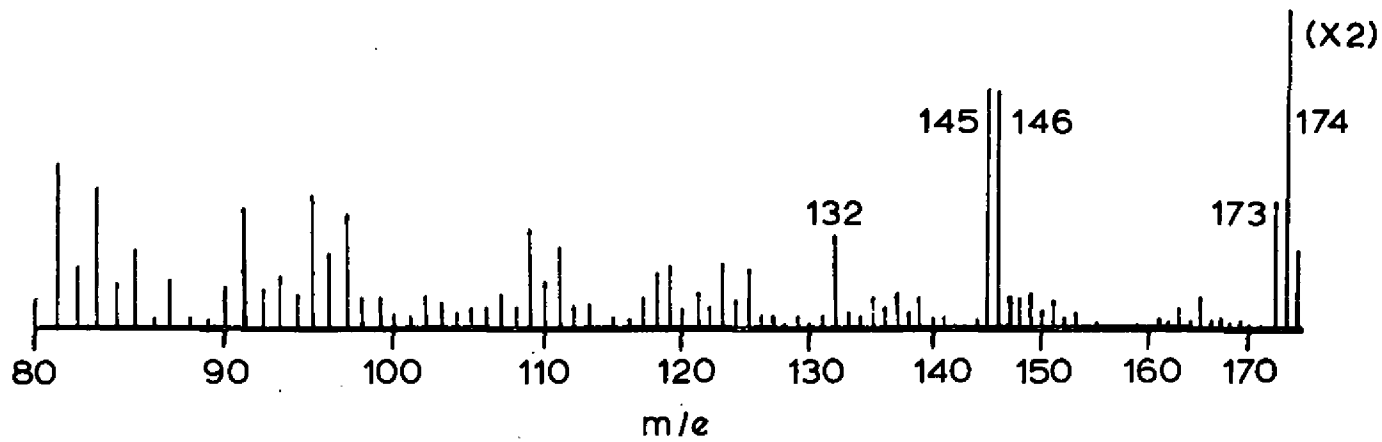
FIG. 3 MASS SPECTRUM OF TDI BY DIRECT INSERTION PROBE

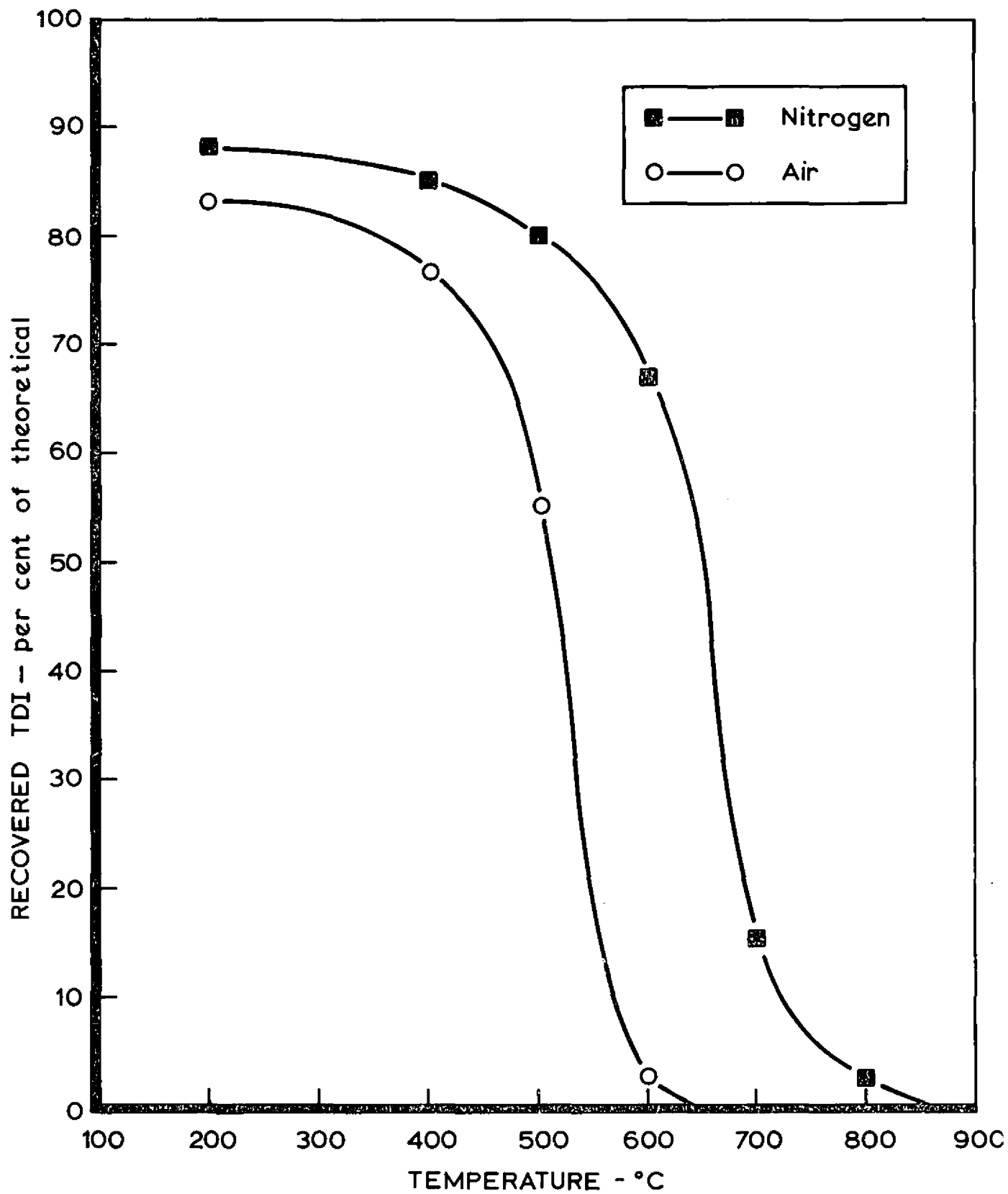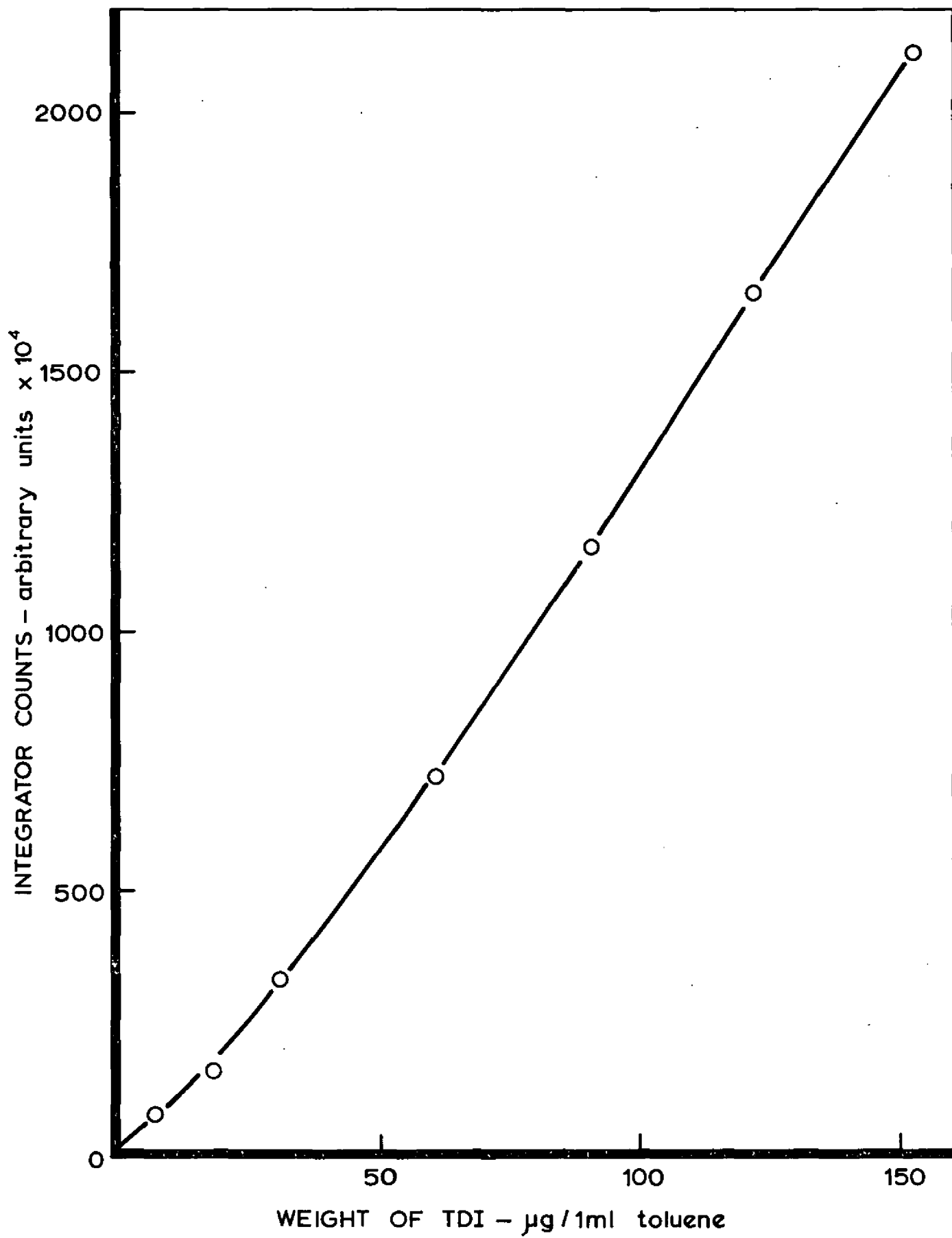FIG. 4   RECOVERY OF TDI IN NITROGEN AND AIR
AS A FUNCTION OF FURNACE TEMPERATURE

FIG. 5 CALIBRATION CURVE FOR TDI
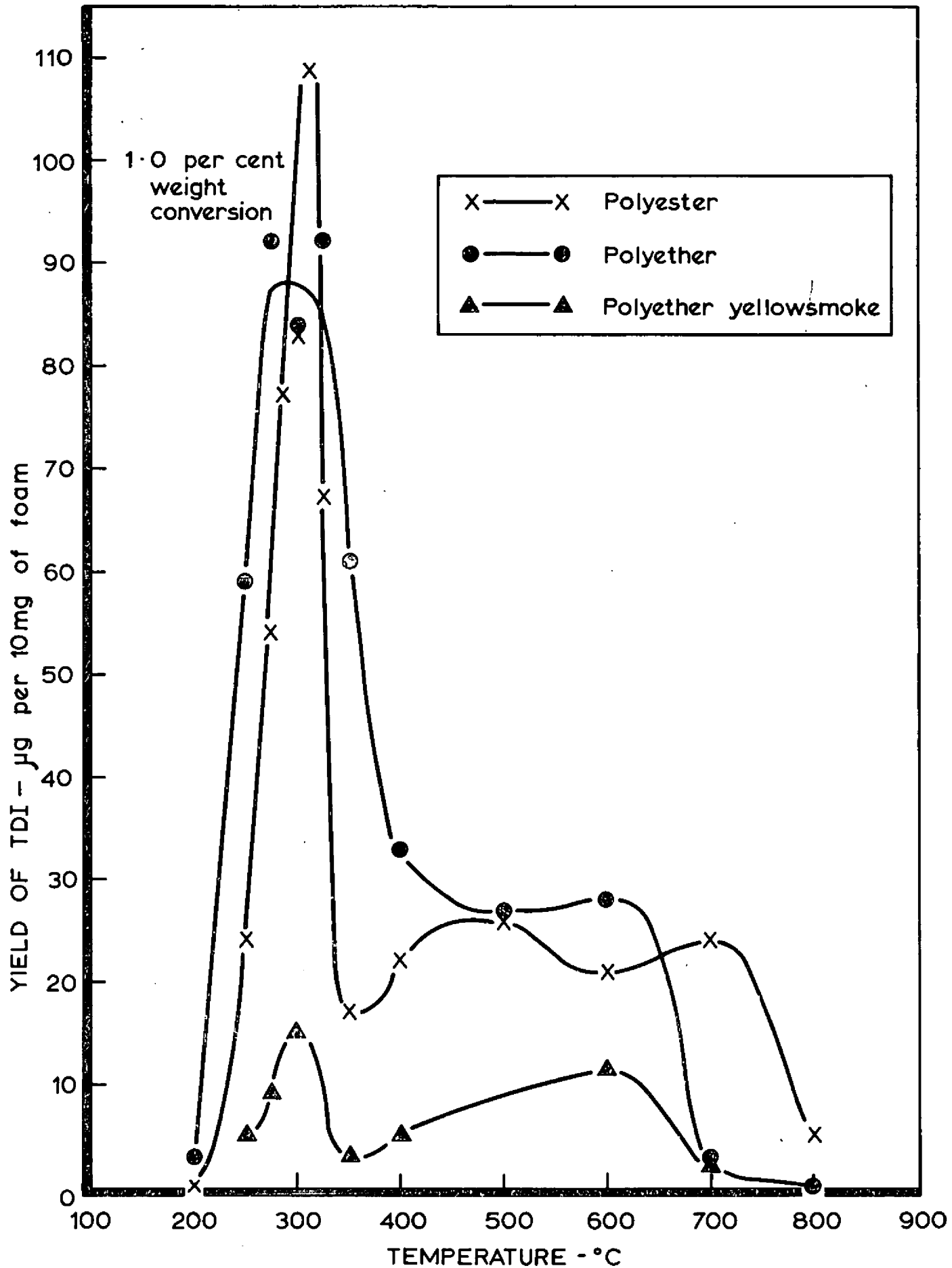
FIG. 6 YIELDS OF TDI FROM THE THERMAL
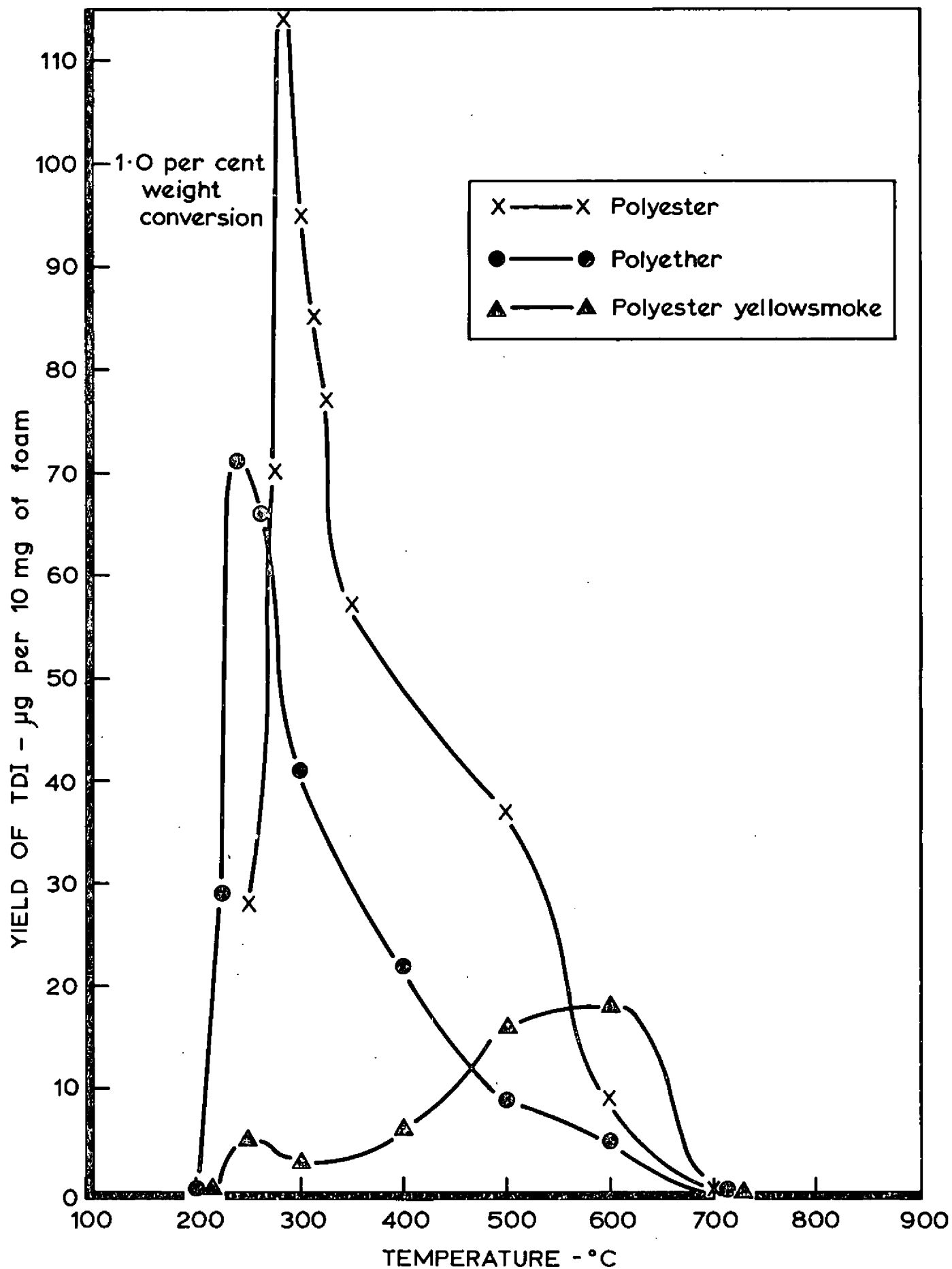DECOMPOSITION OF POLYESTER AND
POLYETHER FOAMS AND YELLOW SMOKE

FIG. 7   YIELDS OF TDI FROM THE THERMAL
DECOMPOSITION IN AIR OF POLYESTER AND
POLYETHER FOAMS AND YELLOW SMOKE