International Standard 6381

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Ethylene and propylene for industrial use — Determination of traces of carbon monoxide and carbon dioxide — Gas chromatographic method

Éthylène et propylène à usage industriel — Dosage des traces de monoxyde et de dioxyde de carbone — Méthode par chromatographie en phase gazeuse

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 6381 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1979.

It has been approved by the member bodies of the following countries :

Australia	Germany, F.R.	Philippines
Austria	Hungary	Poland
Belgium	India	Portugal
China	Italy	Romania
Czechoslovakia	Korea, Rep. of	South Africa, Rep. of
France	Netherlands	Switzerland

No member body expressed disapproval of the document.

Printed in Switzerland

Ethylene and propylene for industrial use — Determination of traces of carbon monoxide and carbon dioxide — Gas chromatographic method

1 Scope and field of application

This International Standard specifies a gas chromatographic method for the determination of traces of carbon monoxide and carbon dioxide in ethylene and propylene for industrial use.

The method is applicable to products having carbon monoxide concentrations greater than 0,1 ml/m^3 and carbon dioxide concentrations greater than 0,5 ml/m^3.

2 Reference

ISO 6377, *Light olefins for industrial use — Determination of hydrocarbon impurities by gas chromatography — General considerations.*

3 Principle

Separation of carbon monoxide, methane and carbon dioxide by gas chromatography through a Carbosieve B or Porapak N column. Passage through a column of hydrogenation catalyst which transforms the oxides of carbon to methane which allows the use of a flame ionization detector to make the final determination.

4 Materials

4.1 Hydrogen, pure and dry.

4.2 Nitrogen, pure and dry.

4.3 Air, compressed, dry.

4.4 Carbosieve B 60-80 or **Porapak N 110-120.**[1)]

Carbosieve B is a special activated carbon which does not show the adsorption phenomena encountered in ordinary activated carbon. It is, however, preferable to avoid prolonged contact with the air and the carrier gas should be dry and oxygen free.

NOTE — The presence of air interferes with the determination of carbon monoxide using Porapak N.

4.5 Chromosorb W AW 80-100.[1)]

4.6 Nickel nitrate hexahydrate [$Ni(NO_3)_2.6H_2O$], analytical grade.

4.7 Nickel based catalyst, prepared *in situ* in the catalyst column as described in the procedure.

NOTE — Other catalysts can be used, for example a reduced ruthenium catalyst.

4.8 Ethylene or **propylene,** pure, free from the impurities to be determined.

4.9 Standard mixture, containing 2 — 10 and 20 ml/m^3 of carbon monoxide and carbon dioxide in pure ethylene or propylene.

5 Apparatus

5.1 Chromatograph, fitted with a **flame ionization detector** and a **gas injection port.**

The gas flow routes are shown in figure 1.

The gases leaving the chromatography column are passed through a catalyst oven before reaching the detector.

5.2 Injection device.

An eight-way valve allows reversal of the gas flow through the chromatography column in order to prevent hydrocarbons containing more than two carbon atoms reaching the catalyst oven, an example of the construction of which is shown in figure 2.

5.3 Columns.

The columns described below are given as examples. Any other columns complying with the same requirements for efficiency may be used.

1) Information on commercially available products can be obtained from the ISO Central Secretariat or from the Secretariat of ISO/TC 47/SC 14 (AFNOR).

5.3.1 Chromatography column

- packing : Carbosieve B or Porapak N
- length : 2 m
- diameter of tube :
  - external 3,1 mm (approximately)
  - internal 2,1 mm (approximately)
- tube material : stainless steel
- apparent density after filling : 0,4 g/cm^3

5.3.2 Catalyst column

- packing : reduced nickel on Chromosorb W
- length : 0,3 m
- diameter of tube :
  - external 6,4 mm (approximately)
  - internal 4,7 mm (approximately)
- tube material : stainless steel
- apparent density after filling : 0,7 g/cm^3

5.4 Detector, flame ionization type.

6 Procedure

6.1 Preparation of the catalyst

Dissolve 49,5 g of the nickel nitrate (4.6) in 100 ml of distilled water and impregnate 50 g of the Chromosorb W (4.5) with this solution. Evaporate the water, fill the catalyst column and install it in the oven. Connect it to the chromatograph (see figure 1) without connecting the detector. Connect the exit end of the column to an aspirator and adjust the flow of hydrogen to 30 ml/min, purge the column for 1 h and then heat the oven to 350 °C and reduce the nickel salt for at least 3 h. Finally raise the temperature of the column to 500 °C and maintain at this temperature for 24 h in order to condition the column.

6.2 Determination

Take into account the information given in ISO 6377.

Pass test portions several times through the chromatograph, using the conditions specified in 6.3, until constant peak heights or areas for the impurities to be determined are obtained. Operate the gas flow valve so as to reverse the flow of carrier gas after the elution of the last impurity being determined.

Pass the standard mixture (4.9) through the chromatograph in the same way.

Identify the peaks on the chromatogram.

6.3 Conditions for chromatography

6.3.1 Temperature

- chromatography column :
  - Carbosieve B : 100 °C,
  - Porapak N : 50 °C;
- catalyst column : 500 °C;
- injector : ambient;
- detector : 125 °C.

6.3.2 Carrier gas

- Hydrogen at a flow rate of 30 ml/min (purified by passing through molecular sieve 5A and soda-asbestos or equivalent absorbent).

6.3.3 Auxiliary gases

- Nitrogen, at 30 ml/min.
- Air, at 300 ml/min.

6.3.4 Quantity injected : 1 ml.

7 Expression of results

See ISO 6377.

8 Test report

See ISO 6377.

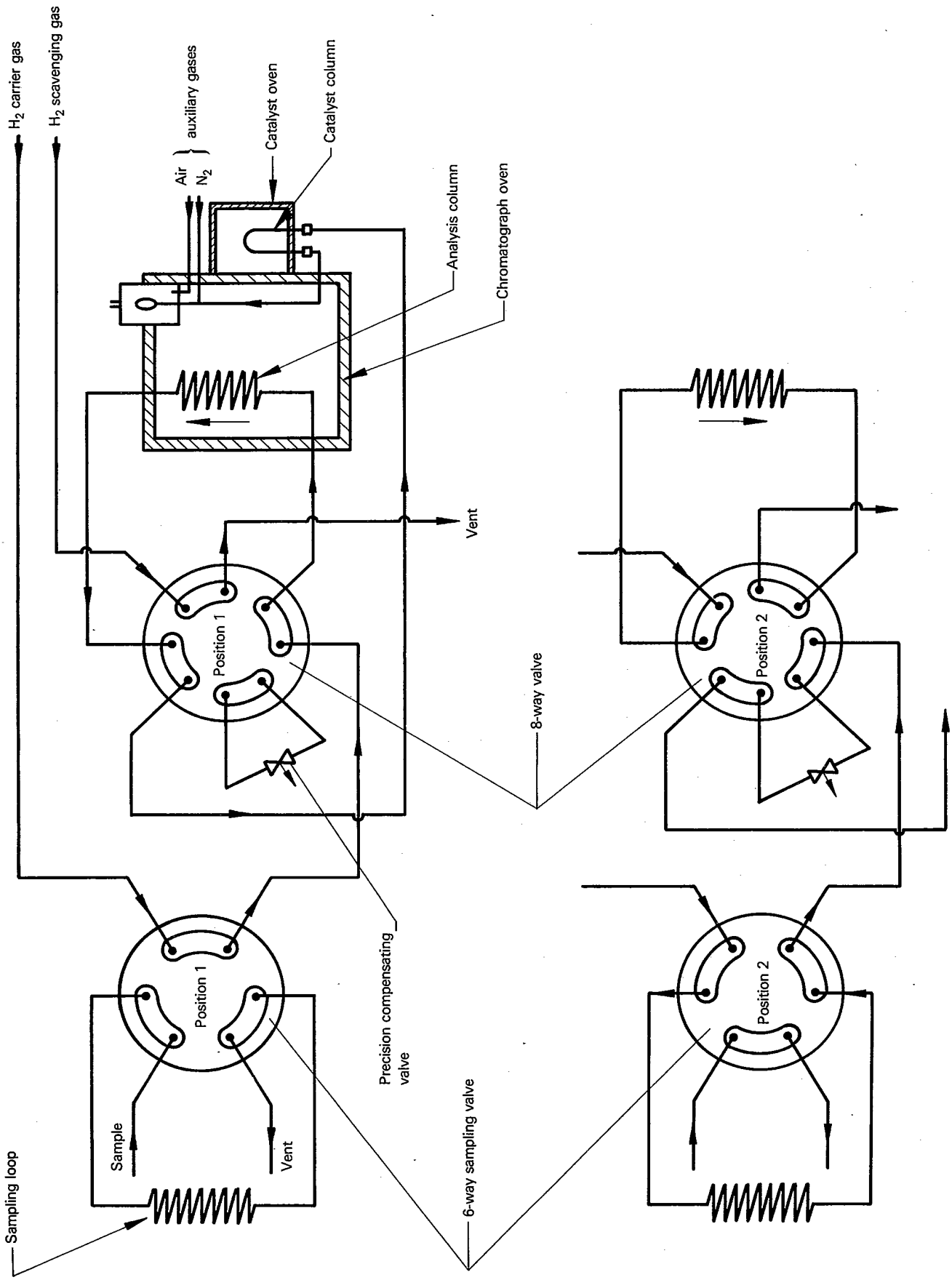
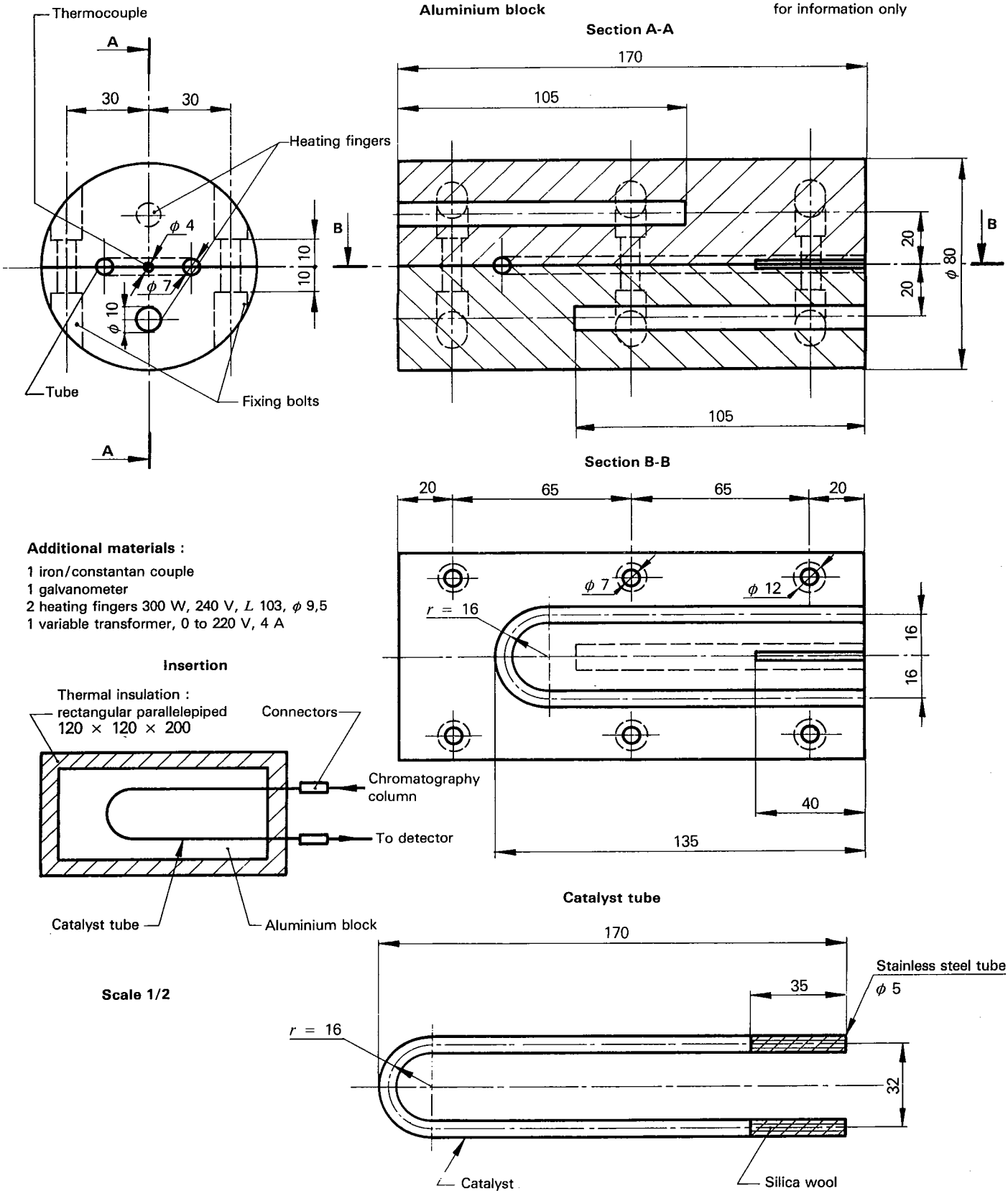


Figure 1 — Apparatus for the determination of carbon monoxide, methane and carbon dioxide in gases

Dimensions in millimetres
for information only



Additional materials :

1 iron/constantan couple
1 galvanometer
2 heating fingers 300 W, 240 V, L 103, ϕ 9,5
1 variable transformer, 0 to 220 V, 4 A

Scale 1/2

Figure 2 — Catalyst oven

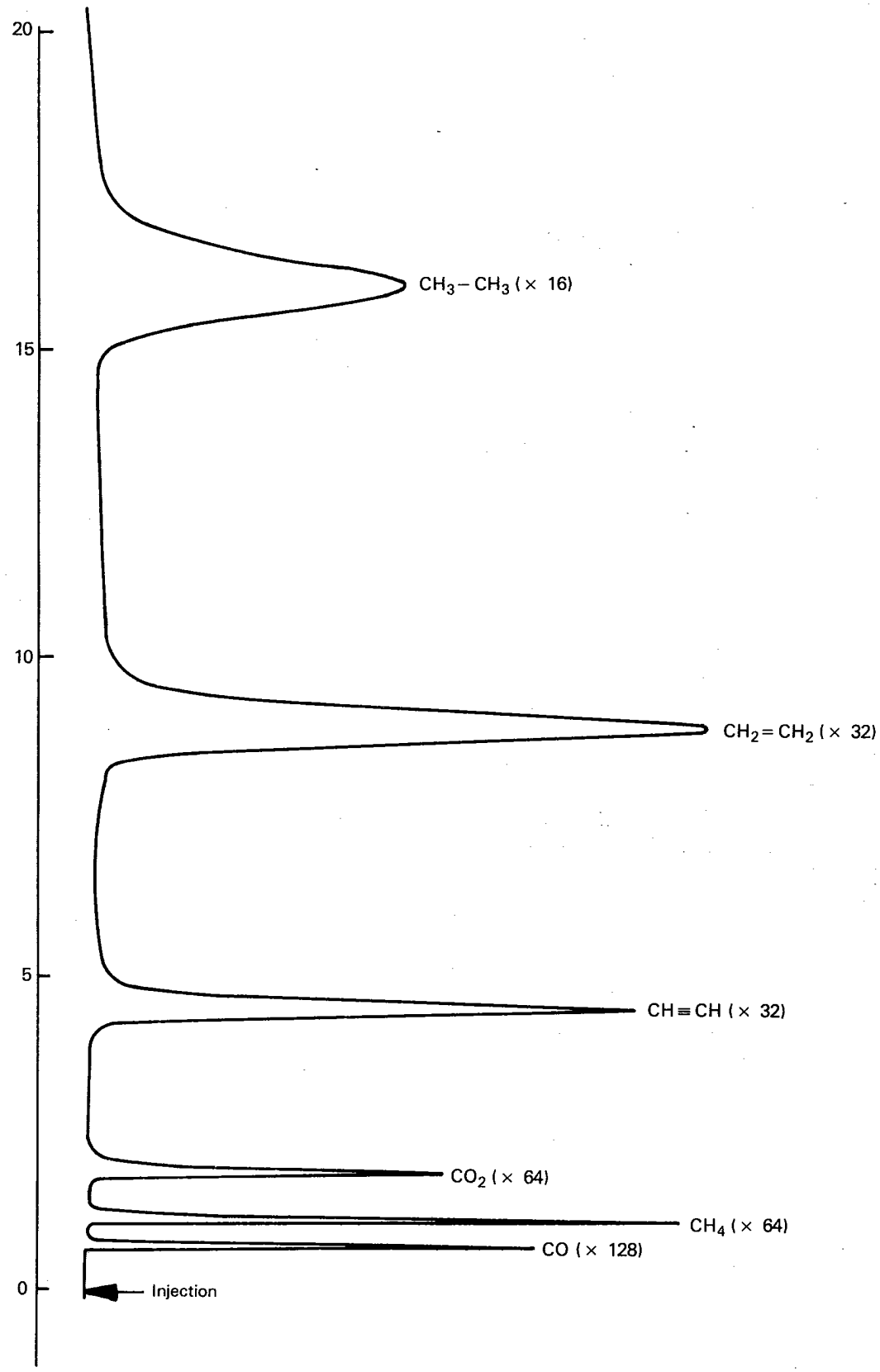


Figure 3 — Typical chromatogram from Carbosieve B column

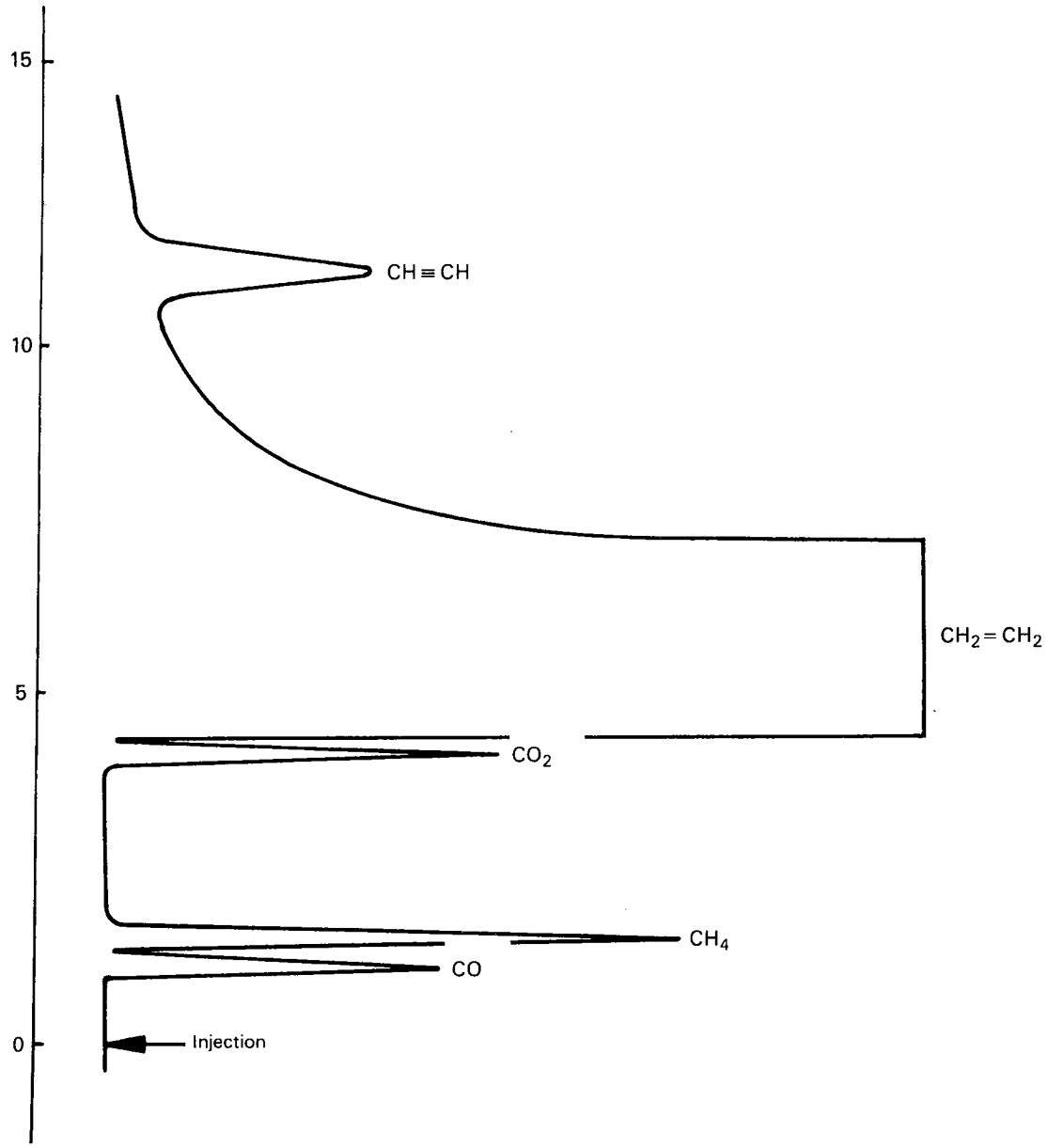


Figure 4 — Typical chromatogram from Porapak N column