Combination of different analytical techniques to determine structure

The determination of the organic structure of an unknown compound is usually achieved by combining the information from several different analytical techniques. This is illustrated by the following worked example for **Compound X**.

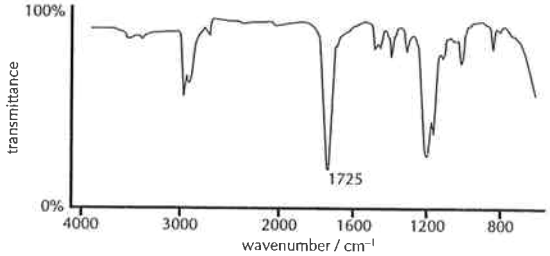
ELEMENTAL ANALYSIS

Compound X was found to contain 48.63% carbon, 8.18% hydrogen, and 43.19% oxygen by mass.

From this information the empirical formula of **Compound X** can be deduced as $C_3H_6O_2$.

Element	Amount / mol	Simplest ratio
C	48.63/12.01 = 4.05	3
H	8.18/1.01 = 8.10	6
O	43.19/16.00 = 2.70	2

INFRARED SPECTROSCOPY

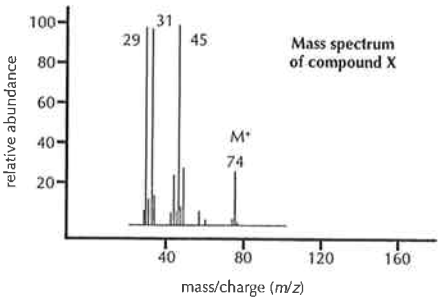


IR spectrum of compound X

Information available from the infrared spectrum:

- Absorption at 2980 cm^{-1} due to presence of C–H in **Compound X**.
- Absorption at 1725 cm^{-1} due to presence of C=O in **Compound X**.
- Absorption at 1200 cm^{-1} due to presence of C–O in **Compound X**.
- Absence of broad absorption at 3300 cm^{-1} indicates **Compound X** does not contain O–H.

MASS SPECTROMETRY



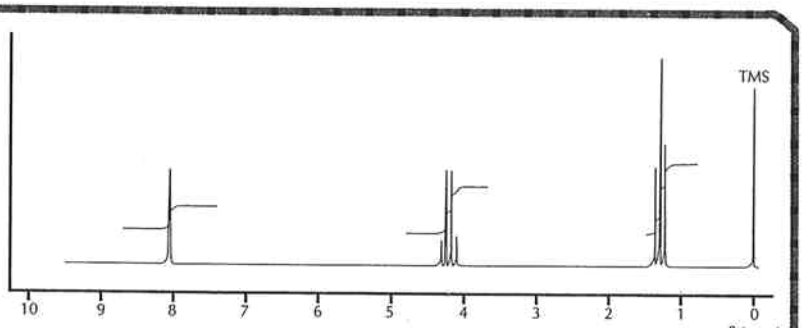
Information available from the mass spectrum:

- Since M^+ occurs at 74 the relative molecular mass of **Compound X** = 74.
- From this and the empirical formula it can be deduced that the molecular formula of **Compound X** is $C_3H_6O_2$.
- Fragment at 45 due to $(M - 29)^+$ so **Compound X** may contain C_2H_5– and/or CHO–.
- Fragment at 31 due to $(M - 43)^+$ so **Compound X** may contain C_2H_5O–.
- Fragment at 29 due to $(M - 45)^+$ so **Compound X** may contain HOOC– or H–C(=O)–O–.
- Peak at 75 due to the presence of ^{13}C.

^{1}H NMR SPECTROSCOPY

Information available from the ^{1}H NMR spectrum:

- Number of separate peaks is three so **Compound X** contains hydrogen atoms in three different chemical environments.
- From the integration trace the hydrogen atoms are in the ratio of 3:2:1 for the peaks at 1.3, 4.2, and 8.1 ppm respectively. Since there are six hydrogen atoms in the molecule this is the actual number of protons in each environment.



^{1}H NMR spectrum of compound X

- From the Data Booklet the chemical shift may be attributed to the following types of proton.

1.3 ppm R–CH_3 (cannot be R–CH_2–R as it is for three protons)

4.2 ppm R–C(=O)–O–CH_2–R

8.1 ppm Not in Data Booklet but consistent with H–C(=O)–O–R.

- From the splitting patterns the number of adjacent hydrogen atoms can be determined.

1.3 ppm	triplet	two adjacent hydrogen atoms
4.2 ppm	quartet	three adjacent hydrogen atoms
8.1 ppm	singlet	no adjacent hydrogen atoms

IDENTIFICATION

All the above information is consistent with only one definitive structure:

Compound X is: H–C(=O)–O–CH_2CH_3
ethyl methanoate