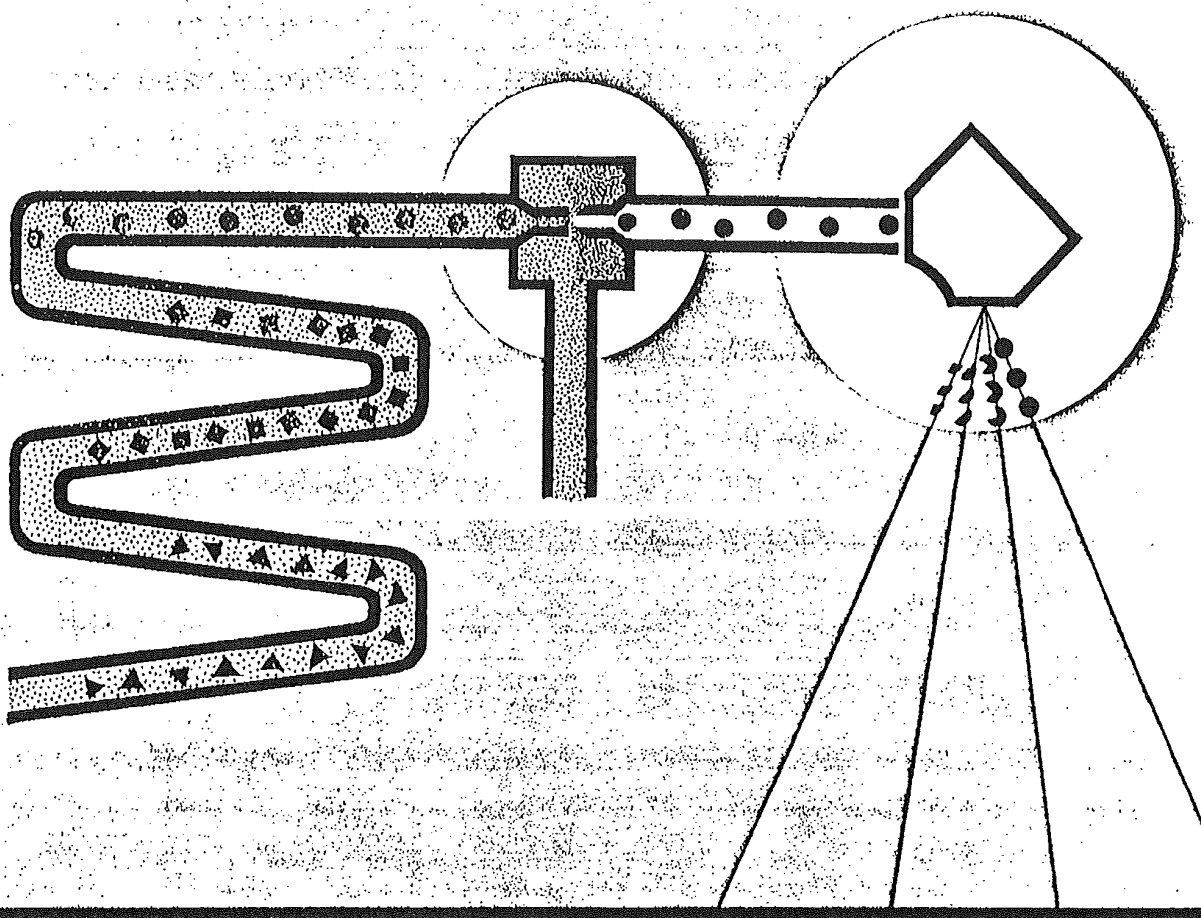


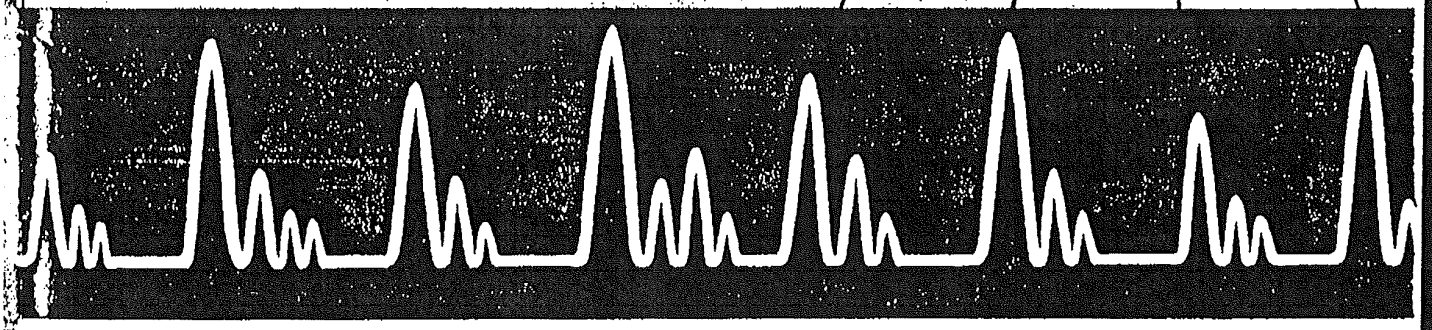
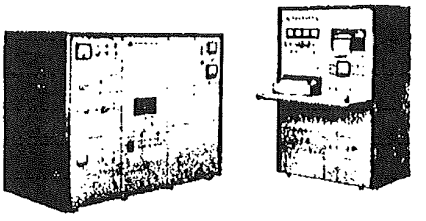


EXHIBIT
WBP M-2

LKB 9000 GAS CHROMATOGRAPH – MASS SPECTROMETER

The complete instrument for gas chromatography and mass spectrometry



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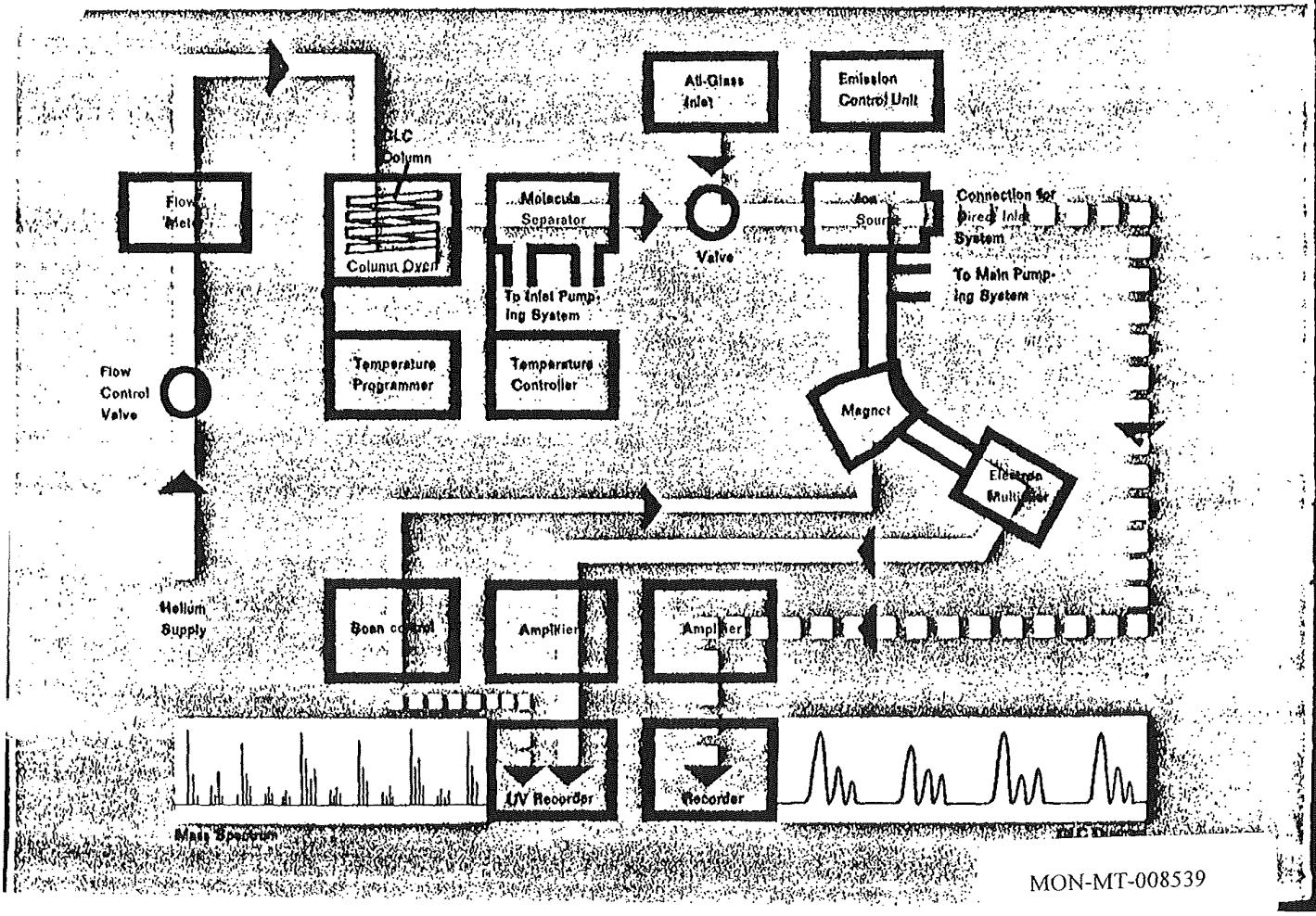
Versatility

The LKB 9000 is a fully equipped combination instrument providing simple, reliable and flexible operation in the routine mass spectrometric analysis of organic substances which have been purified by the built-in gas chromatograph. It utilizes the most advanced and sophisticated methods and instrumentation for each of its important unit operations, from sample introduction into the gas chromatograph to recording of a mass spectrum. Alternative inlet systems are provided for operation of the mass spectrometer with previously purified liquids and solids. We are justly proud of the performance of this first commercial instrument to integrate a gas chromatograph, a molecule separator and a mass spectrometer into one unit, an instrument which is eminently suitable for:

- **Biochemical Analysis**
- **Identification of Trace Components in Complex Mixtures**
- **Elucidation of Molecular Structures**
- **Studies of Isotope Abundance**

Block diagram of LKB 9000 gas chromatograph – mass spectrometer.



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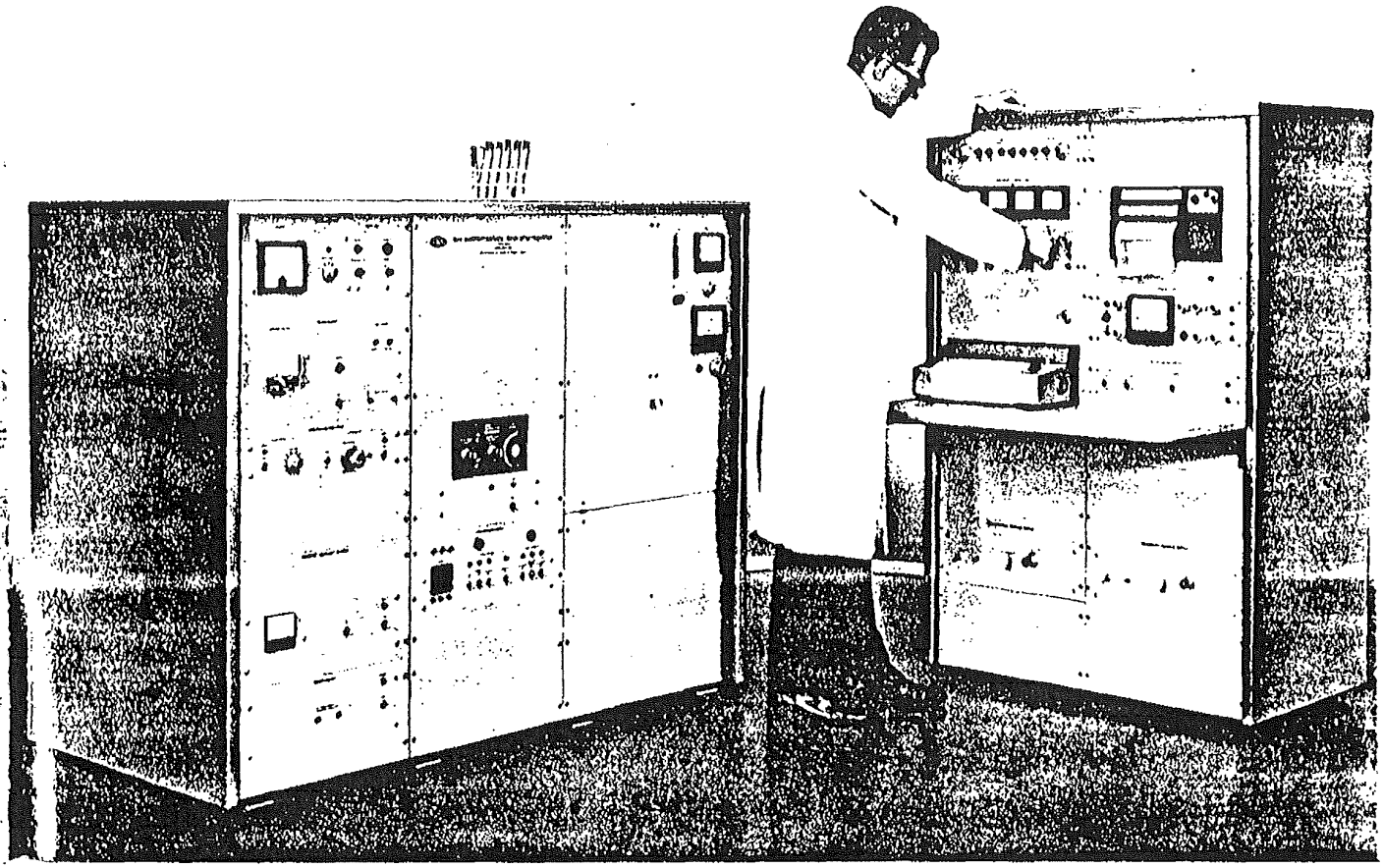


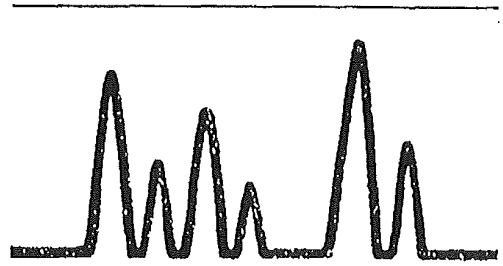
LKB 9000 showing the analyzer unit (on left) and the control unit.

Advanced instrumentation

GAS CHROMATOGRAPHY for the preliminary purification of organic compounds in complex mixtures is achieved with coiled columns housed in a compact oven. A circulating air bath assures accurate temperature regulation in isothermal or temperature-programmed operation.

SAMPLE ENRICHMENT in a unique molecule separator*, is carried out by the preferential removal of carrier gas from the effluent stream of the gas chromatograph.

IONIZATION is achieved by electron impact. Automatic regulation of electron energy provides optimal levels of ionization during operation of the ion source as a GLC detector, and for the reproducible production of an array of fragment ions during recording of a mass spectrum.
Mass spectra are obtained with a rapid-scanning, single-focusing magnetic sector analyzer with sufficient resolving power for operation with compounds in a molecular weight range up to 1200.
Optimal resolving power under different operating conditions is made possible with externally variable ion source and collector slits.



* According to Becker, Kernforschungszentrum, Karlsruhe, and Ryhage, Karolinska Institutet, Stockholm.

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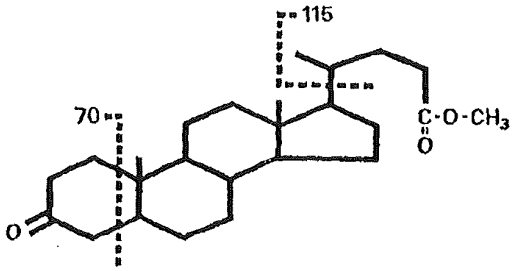
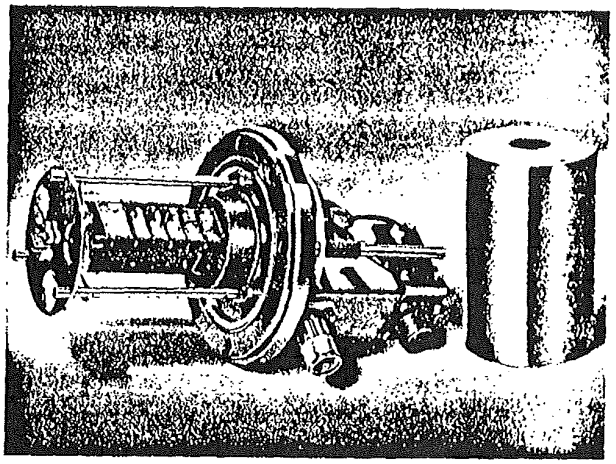
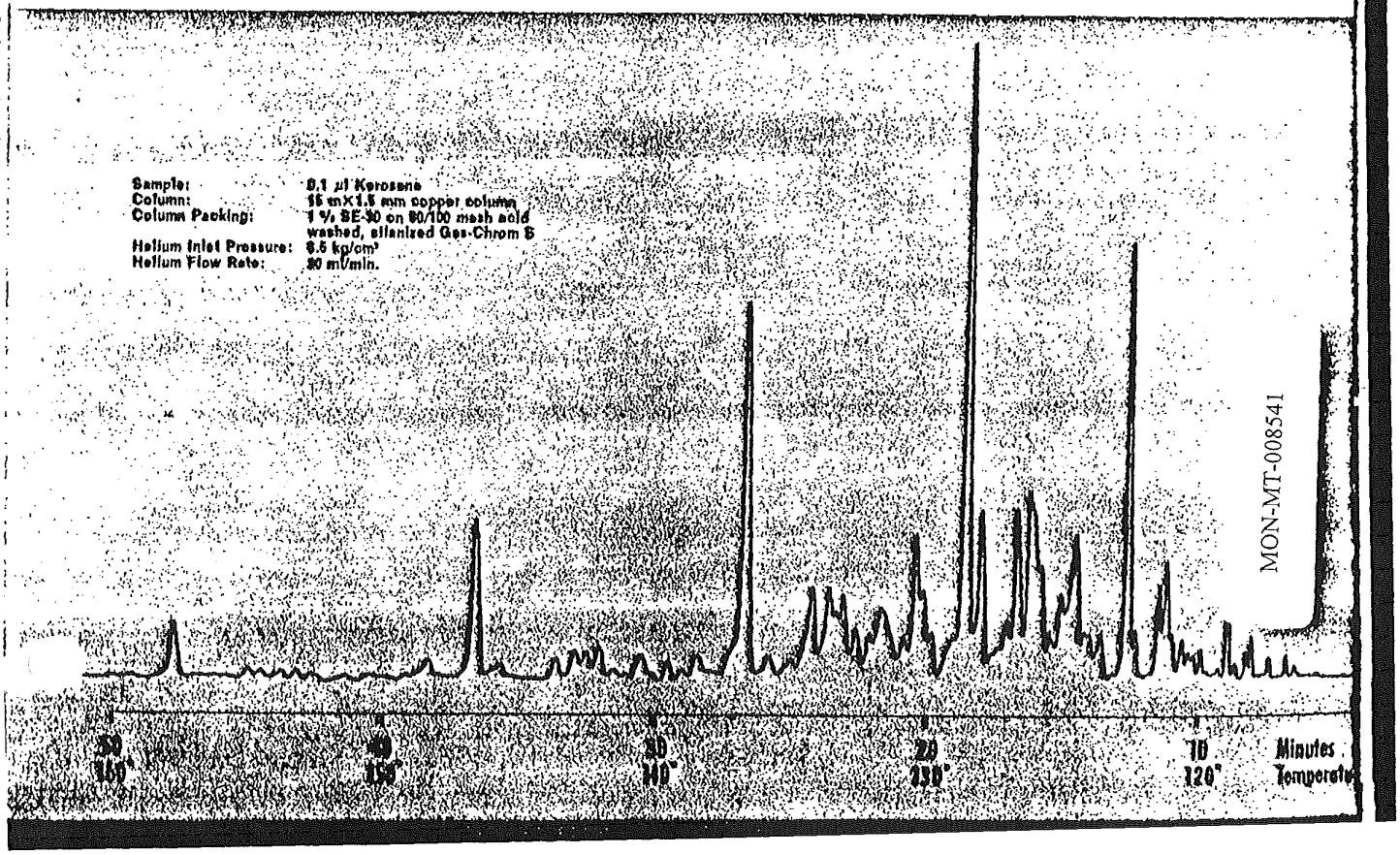
Gas Chromatography

Components for the GLC inlet are designed to provide optimal performance with the most demanding chromatographic conditions. Minimum dead volume and effective uniform heating of all parts ensure symmetrical peaks with no loss of column efficiency, even with capillary columns and long packed columns. This is well illustrated by the chromatogram on this page which shows the separation of hydrocarbons from Kerosene in a temperature programmed analysis on a 1 % SE-30 column 15 meters in length.





Electron Multiplier Assembly



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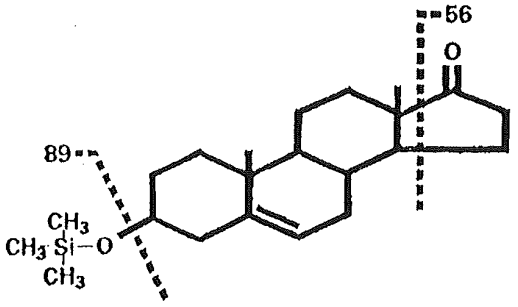
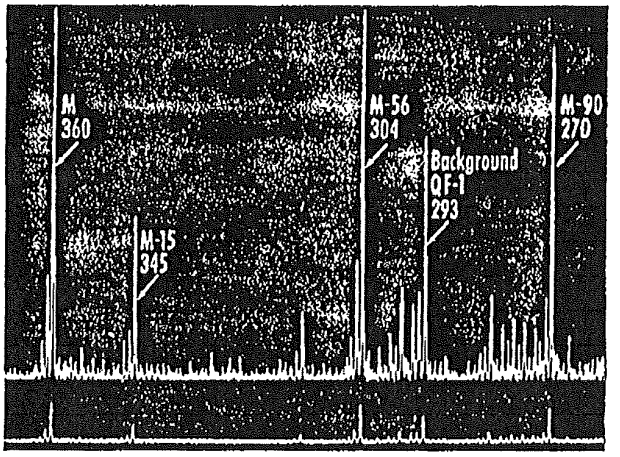
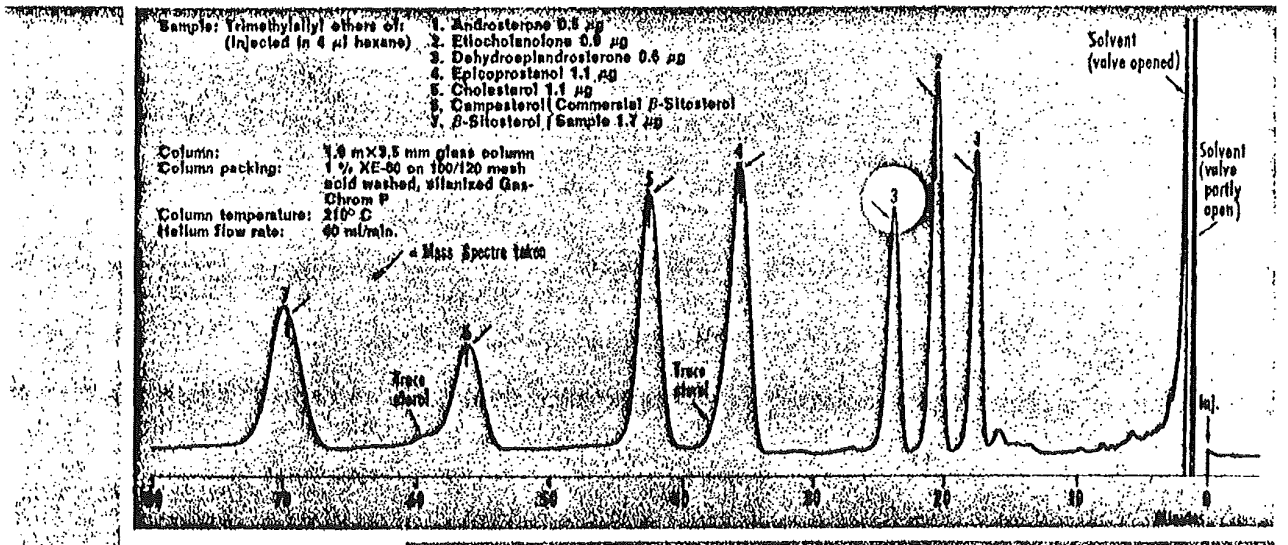
High Sensitivity

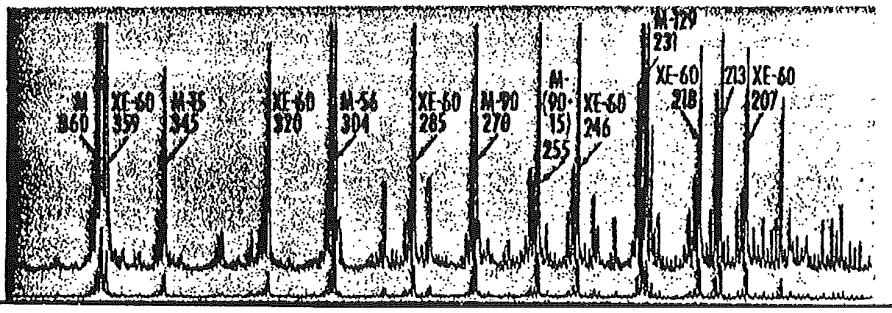
Due to the fact that minute samples may be used (0.01 microgram with a capillary column or 0.1 microgram with a packed column is sufficient for a complete mass spectrum), the LKB 9000 is ideal for studies of analytical problems in biochemistry. It is eminently suitable for the analysis of amino acid and carbohydrate derivatives, steroids and bile acid esters, insecticides and for studies of lipid metabolism. Mass spectra of biologically important high molecular compounds are easily obtained following purification with the GLC inlet. With this inlet, a resolving power of more than 600 can be attained. Under ideal operating conditions using an all-glass or direct probe inlet M/ΔM is 1000 at ion-source exit slit and collector slit settings of 0.05 and 0.10 mm respectively (10 % valley definition).





Fast scan (m/e 24–400 in 1 sec.) of 0.5 μg dehydroepiandrosterone trimethylsilyl ether.





Upper end of the mass spectrum taken at peak 3 of the above GLC diagram.

Electron energy: 70eV

Scan speed: m/e 12–400 in 4 sec.

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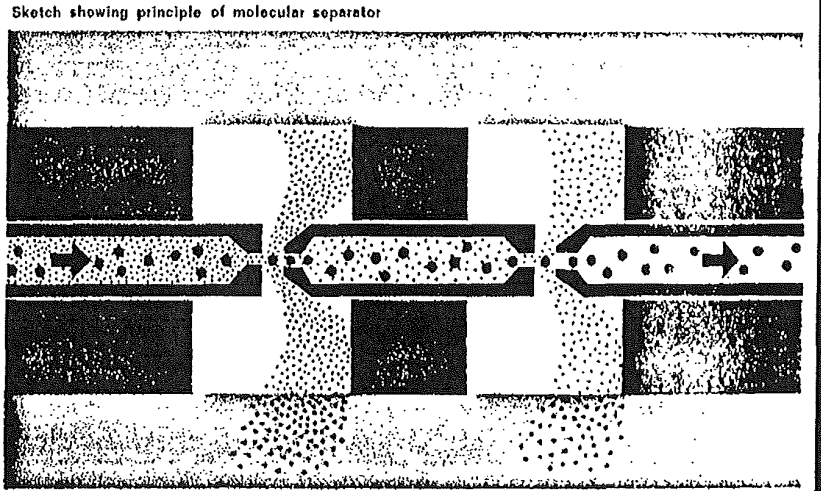
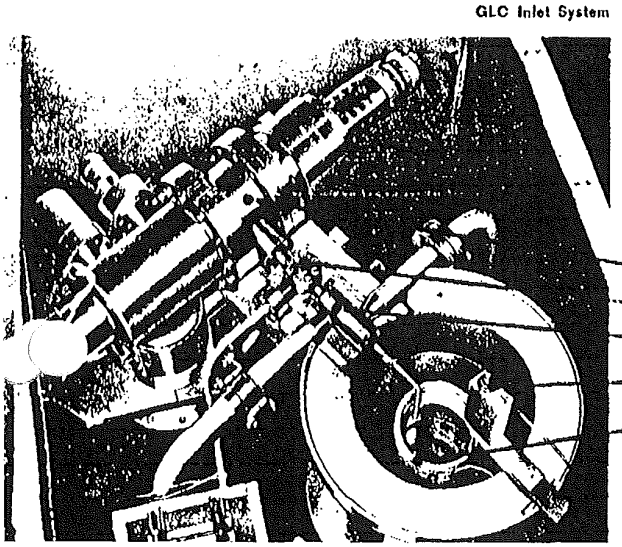
Sample Enrichment

Substances eluted from the GLC column are carried along with the helium carrier gas into the molecule separator, which, in two stages, preferentially removes most of the helium, while the sample passes into the ion source chamber in a highly concentrated molecular beam. The jet-type separator, of Becker-Ryhage design, is equipped with forevacuum and diffusion pumps. The unit provides a pressure drop from approximately atmospheric to about 10^{-4} mm Hg, and is effective with organic compounds of all molecular weights. The large pressure drop in the molecule separator enables operation of the spectrometer at less than 10^{-6} mm Hg. Independent, controlled heating of the separators prevents sample condensation.

Sketch showing principle of molecular separator



GLC Inlet System



Accessibility

Compact arrangement of chromatographic column, molecule separator, valve, and ion source ensures easy operation and negligible zone widening in the effluent prior to ionization.

Other Inlets

Convenient arrangement of valves facilitates quick switching between the GLC inlet and the standard all-glass inlet. Provision is made for connection of a direct probe and vacuum lock system.

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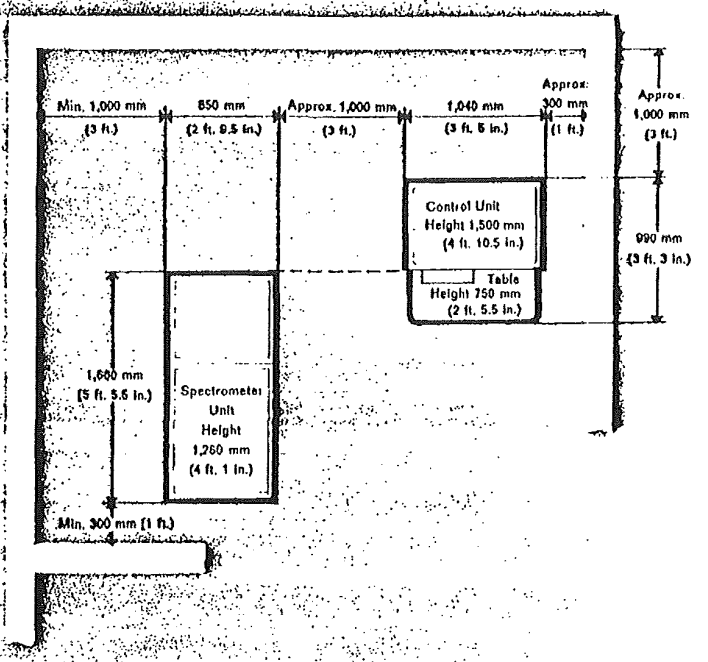
Space Diagram



Technical Specifications

The LKB 9000 is a single focusing mass spectrometer, equipped with a 60° sector, 20 cm radius magnetic analyzer and a sweep generator for fast scanning of spectra. The ion source is of the electron bombardment type, utilizing a rhenium filament and is operated at a variable accelerating voltage of 3.5 kV maximum. The system for measurement of ion intensity consists of a 14-stage electron multiplier, followed by an electrometer, and a wide band amplifier feeding a direct-writing UV recording oscillograph.

The high capacity vacuum system consists of two isolated pumping assemblies, one for the analyzer tube and one for the inlet systems. A cold trap is provided for liquid N_2 or CO_2 but this need only be used for mass spectral analysis of extremely small samples and during bake-out.

Mass range

Automatic scanning: Continuously variable m/e 2-1200
Manual adjustment: Continuously variable m/e 2-700 (at 3.5 kV accelerating voltage).

Resolution

$M/\Delta M$ = 1000 at ion source exit slit and collector slit settings of 0.05 and 0.10 mm respectively (10 % valley definition).

Sensitivity

At optimal performance the required sample amount is less than 0.01 µg (capillary column) and 0.1 µg (packed column).

Scanning

Magnetic scan, the range of which can be pre-set to any suitable value by means of limit switches. Scanning time 1—960 seconds in 9 steps for full scan.

Examples: m/e 12— 100 in 2.5 sec.
m/e 12— 400 in 4.2 sec.
m/e 24— 800 in 4.2 sec.
m/e 48—1000 in 3.6 sec.
} $M/\Delta M$ = 1000

m/e 12— 100 in 1.0 sec.
m/e 12— 400 in 1.7 sec.
m/e 24— 800 in 1.7 sec.
m/e 48—1000 in 1.5 sec.
} $M/\Delta M$ = 400

Accelerating voltage

2.5, 3.0 and 3.5 kV using 3-step selector. Stability better than 0.01 % per 10 min.

Magnetic field

0—13 kilogauss, continuously variable. Stability better than 0.01 % per 10 min.

Electron energy

10—100 eV. Automatic step-up to pre-selected value during scan (e.g. from 20 to 70 eV).

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Sensitivity 0.1 µg (packed column)

Ion source exit slit

Continuously adjustable 0—0.4 mm from outside the vacuum.

Collector slit

Continuously adjustable 0—0.6 mm and rotatable ± 2° from outside the vacuum.

Electron multiplier

14 dynodes, variable gain 10^8—10^7. The multiplier is fed by a high-voltage D.C. power supply (stability better than 0.01 % per 10 min.), 1.7—3.7 kV in eleven steps.

Recording

A collector electrode, located just after the exit slit of the ion source, is connected through an electrometer amplifier to a strip-chart recorder. With GLC, typical chromatograms are produced by continuous recording of total ion current in the spectrometer. With other inlet systems the recorder serves to indicate optimal ion intensity for mass spectral analysis.
Mass spectra are recorded at 3 sensitivities on a multichannel UV recording oscillograph, the operation of which is automatically synchronized with the automatic scan.

Inlet systems

Gas chromatograph and all-glass system for alternative use. Provision made for connection of optional inlet systems.

Gas chromatograph

The cylindrical oven with a circulating air bath accepts coiled columns of variable length but maximum dimensions of 140 mm (5½") coil diameter and 100 mm (4") heights. Column temperatures are regulated by proportional control (25—350°C) for isothermal operation, and can be programmed linearly at rates up to 15°C per min. A carrier gas flow meter and flow control regulator are included with the standard instrument.

Molecule separator

2-step jet separator of the Becker-Ryhage type, separately heated by means of a built-in temperature controller. Temperature range 50—300°C.

The enrichment factor is greater than 100 when working with compounds in the molecular weight range above 400.

The principles of the molecule separator are protected by patents in U.S.A., West Germany, France, Great Britain, Italy, Canada, and Japan.

Vacuum system

Analyzer tube:	Fore pump	2.5 m³/h
	Oil diff. pump	550 ltrs/sec.
	Mercury booster pump	12 ltrs/sec.

Cold trap for liquid N_2 or CO_2

Ultimate vacuum 10^{-7} mm Hg (closed inlet valve)

Inlet systems:	Fore pump	9.5 m³/h
	Oil diff. pump	150 ltrs/sec.
	Mercury booster pump	2 ltrs/sec.
Gauges:	Pirani and Penning type vacuum gauges.	

Bake-out

Analyzer tube and other high vacuum parts can be outgassed at 300—400°C by indirect heating.

Mains connection

220/380V (220V to ground, 380V between phases) 3-phase A.C. Other voltages are suitable, but require a separate transformer, optionally available.

Power consumption

Approx. 8kVA, unsymmetrical load, max. 15A/phase (at 220/380V).

Cooling water consumption

Approx. 3 ltrs/min., temperature max. 20°C (70° F).

Dimensions and weights

Analyzer unit:	h = 125 cm (4 ft. 1 in.)
	w = 85 cm (2 ft. 9 in.)
	l = 165 cm (5 ft. 5 in.)
	Approx. weight = 600 kg (1,300 lb)
Control unit:	h = 150 cm (4 ft. 11 in.)
	w = 105 cm (3 ft. 5 in.)
	l = 105 cm (3 ft. 5 in.)
	Approx. weight = 250 kg (550 lb)

LKB

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