CHAPTER 2

EXPERIMENTAL PROCEDURE

2.1 Chemical reagents and equipments

2.1.1 Chemical reagents

1) Ammonium metatungstate hydrate, $(NH_4)_6H_2W_{12}O_{40}.xH_2O$, MW = 2,956.30, $\geq$ 99.0%, Fluka
2) Hexadecyltrimethylammonium bromide (CTAB), $C_{19}H_{42}BrN$, MW = 364.46, 98%, Fluka
3) Sodium tungstate dihydrate, $Na_2WO_4.2H_2O$, MW = 329.86, 99-101.0%, Carlo Erba, Italy
4) Ammonium sulphate, $(NH_4)_2SO_4$, MW = 132.13, 99%, Ajax
5) Hydrochloric acid, HCl, MW = 36.46, min 37.5%, Merck, Germany
6) Ethanol, C_2H_5OH, 95%, Merck, Germany
7) Deionized water

2.1.2 Equipments

1) Hotplate and magnetic stirrer, model 502P-2, PMC Industries, Inc., San Diego, U.S.A.

2) Analytical balance, Model BP-210S, Satorius AG. Goettingen, Germany

3) Oven, Model UE-400, Memmert, Germany

2.2 Synthesis methods

2.2. 1 Synthesis of tungsten oxide by a hydrothermal method using ammonium metatungstate hydrate as a tungsten source

1.38 g of $(NH_4)_6H_2W_{12}O_{40}.xH_2O$ was dissolved in 20 ml deionized water; 0, 2.50, 5.00 and 7.50 ml 1M HCl was subsequently added with 30 min continuous stirring. 0.36 g of CTAB surfactant was added to each solution with an additional 30 min of continuous stirring. The reaction hydrothermally proceeded at 200 °C for 24 hours to form precipitates, and left to naturally cool to room temperature. Finally, light-green precipitates were synthesized, separated by filtration, washed with deionized water and ethanol, and dried at 70 °C for 12 h.

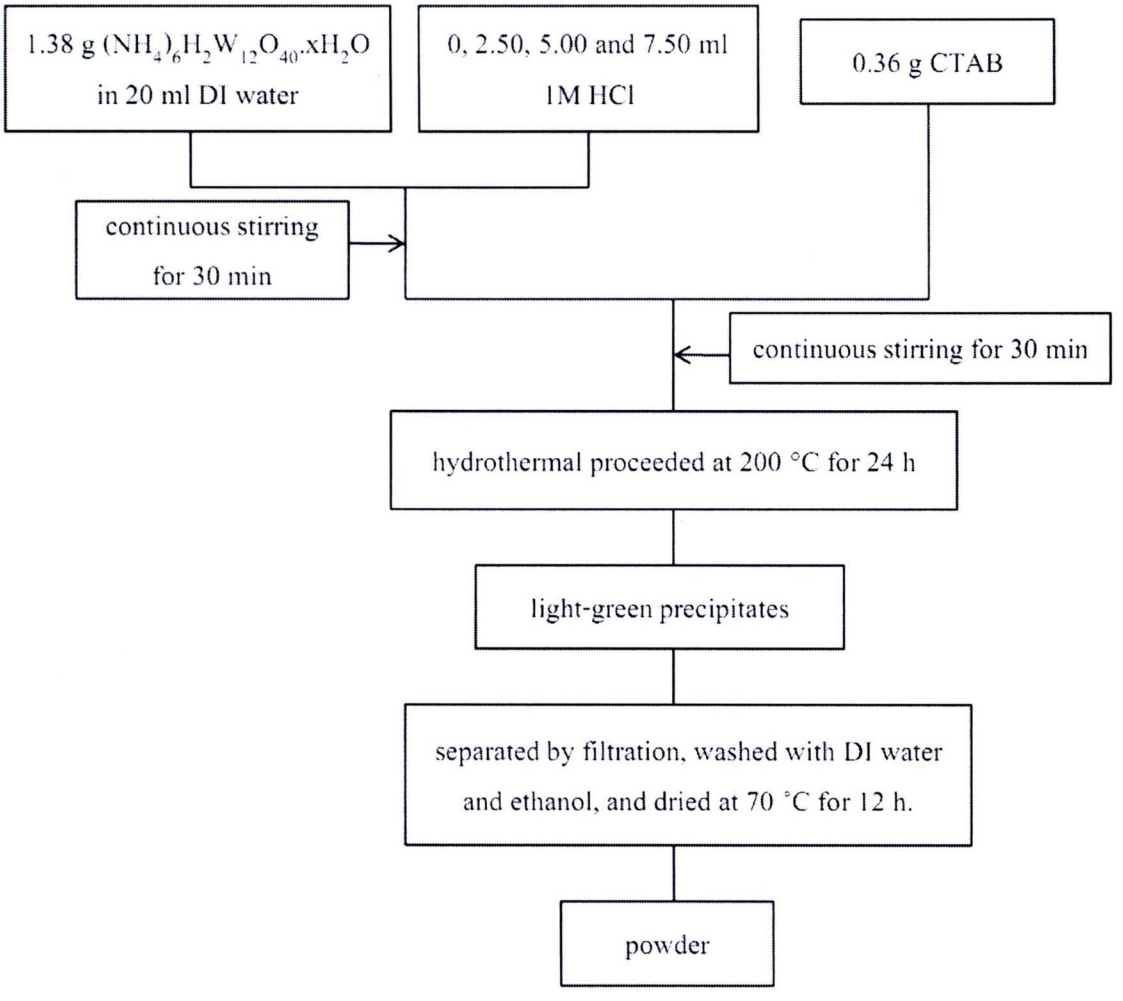

Figure 2.1 Schematic diagram used for preparing tungsten oxide by a hydrothermal method using ammonium metatungstate hydrate as a tungsten source with different volumes of 1M HCl

2.2. 2 Synthesis of tungsten oxide by a hydrothermal method using sodium tungstate dihydrate as a tungsten source

2.00 g of $Na_2WO_4.2H_2O$ was dissolved in 35 ml deionized water, 3M HCl was subsequently added with 30 min continuous stirring. 2.00 g of $(NH_4)_2SO_4$ was added to each solution with an additional 10 min of continuous stirring. The reaction hydrothermally proceeded to form precipitates, and left to naturally cool to room temperature. Finally, light-green precipitates were synthesized, separated by filtration, washed with deionized water and ethanol, and dried at 70 °C for 12 h.

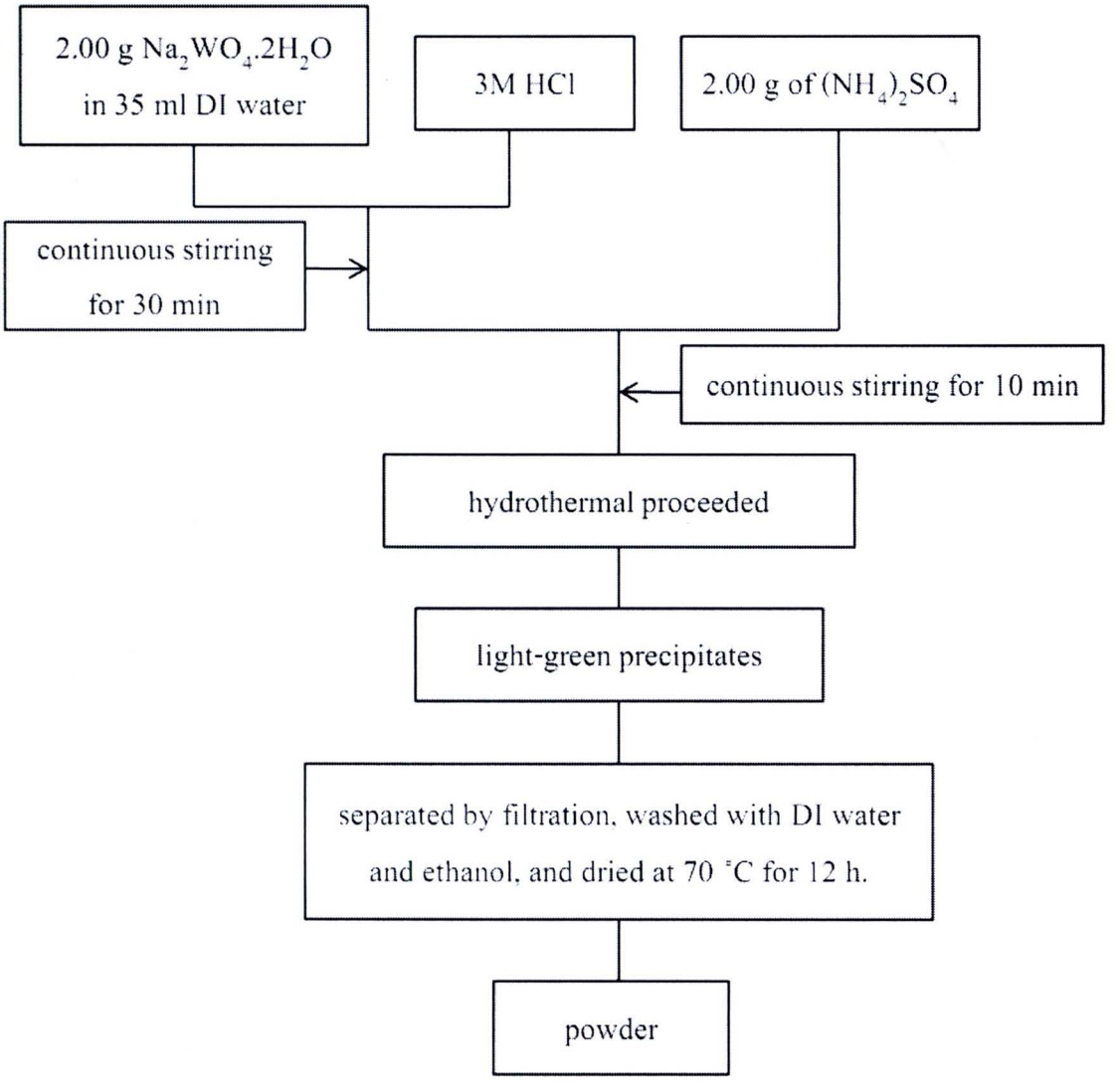

Figure 2.2 Schematic diagram used for preparing tungsten oxide by a hydrothermal method using sodium tungstate dihydrate as a tungsten source with different volumes of 3M HCl, length of reaction times and reaction temperature

Table 2.1 Experimental conditions for synthesizing of tungsten oxide by a hydrothermal method using sodium tungstate dihydrate as a tungsten source with different volumes of 3M HCl, length of reaction times and reaction temperature.

Condition		3M HCl (ml)	Temperature (^{o}C)	Time (h)
Effect of acidity	A1	4.00	180	24
	A2	4.50	180	24
	A3	5.00	180	24
	A4	5.50	180	24
	A5	6.00	180	24
	A6	6.50	180	24
	A7	7.00	180	24
Effect of reaction temperature	B1	5.00	120	12
	B2	5.00	140	12
	B3	5.00	160	12
	B4	5.00	180	12
	B5	5.00	200	12
Effect of reaction time	C1	5.00	200	0
	C2	5.00	200	6
	C3	5.00	200	12
	C4	5.00	200	24
	C5	5.00	200	48
	C6	5.00	200	72

2.3 Characterization

2.3.1 X-ray diffraction (XRD)

The crystallinity and phase purity of the products were analyzed by using X-ray diffractometry (XRD) with Cu K_α radiation (λ = 1.5418 Å) operating at 20 kV-15mA, at a scanning rate of 5°/min in the 2θ range of 10°-60°. The identification samples were assisted by Philips X'Pert Highscore Computer Software (search-match program) on the database of JCPDS software [29].

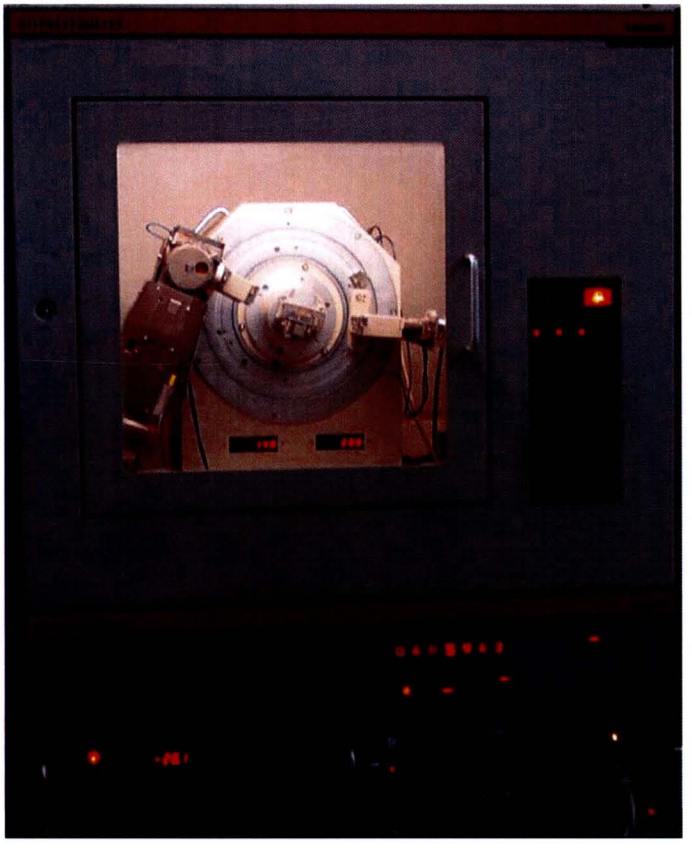

Figure 2.3 X-ray diffractometer

2.3.2 Fourier transform inferred (FTIR) spectroscopy and Raman spectroscopy

The vibration modes of products were analyzed by Fourier transform inferred (FTIR) and Raman spectroscopy. The samples were diluted 40 times by KBr and operated in the wavelength range 400-4000 cm^{-1} for FTIR analysis (BRUKER TENSOR27). Raman spectroscopy was operated on HORIBA JOBIN YVON T64000 Raman spectrometer with 50 mW and 514.5 nm wavelength Ar laser.

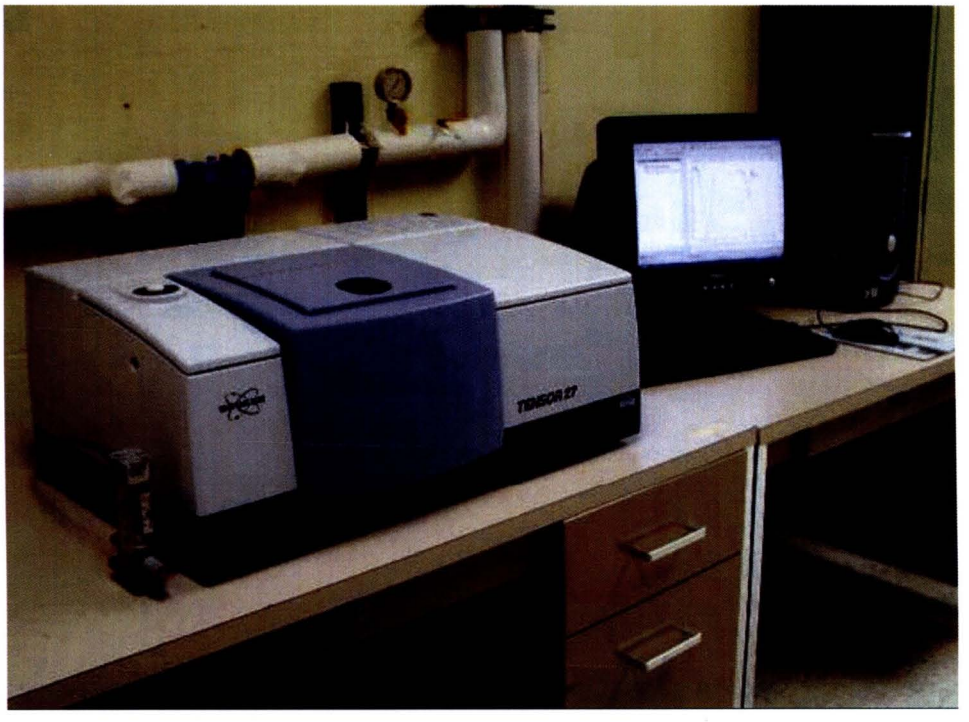

Figure 2.4 Fourier transform inferred spectroscopy

Figure 2.5 Raman spectroscopy

2.3.3 Field Emission Scanning Electron Microscopy (FE-SEM)

The morphology of the products were analyzed by Field Emission Scanning Electron Microscopy, JEOL model SEM, JSM-6335F operating at 15 kV as a accelerating voltage. The products were dispersed in absolute ethanol using an ultrasonic bath. The dispersed samples were dropped on conductive gold tape which attached to the SEM stubs. The stubs were then coated with gold particles by plasma sputtering under argon atmosphere in order to increase conductivity.

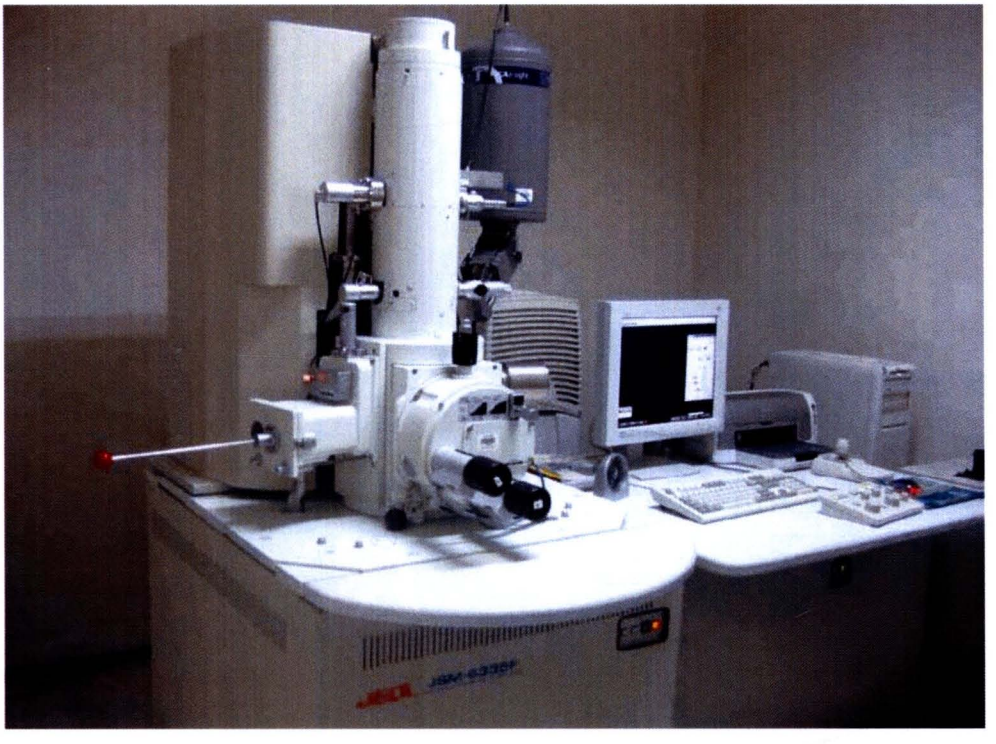

Figure 2.6 Field Emission Scanning Electron Microscopy

2.3.4 Transmission Electron Microscopy (TEM)

The morphology and structure of products were also characterized by transmission electron microscope, JEOL model JEM-2010 operating at 20 kV. The samples for TEM analysis were prepared by dispersing their small amount in absolute ethanol and put a drop of the solutions onto copper grids coated with holey carbon films and letting the ethanol evaporate slowly in ambient atmosphere.

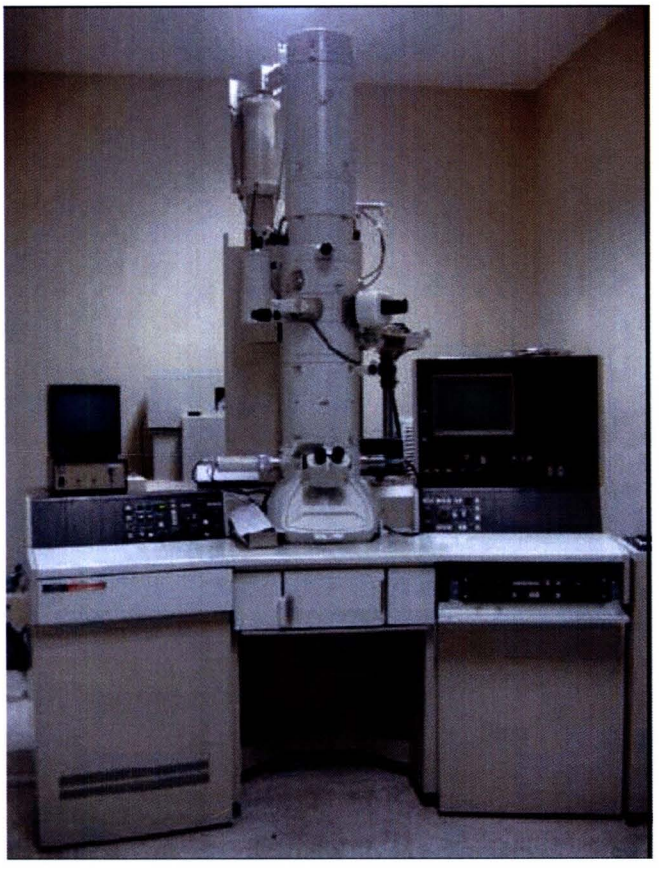

Figure 2.7 Transmission Electron Microscope

2.3.6 Photoluminescence Spectroscopy

The photoluminescence properties of products were investigated by Perkin Elmer Luminescence spectrometer LS50B at room temperature using an excitation wavelength of 200 nm. The appropriated amount of powder samples were dispersed in absolute ethanol using ultrasonic bath, and tested for emission.

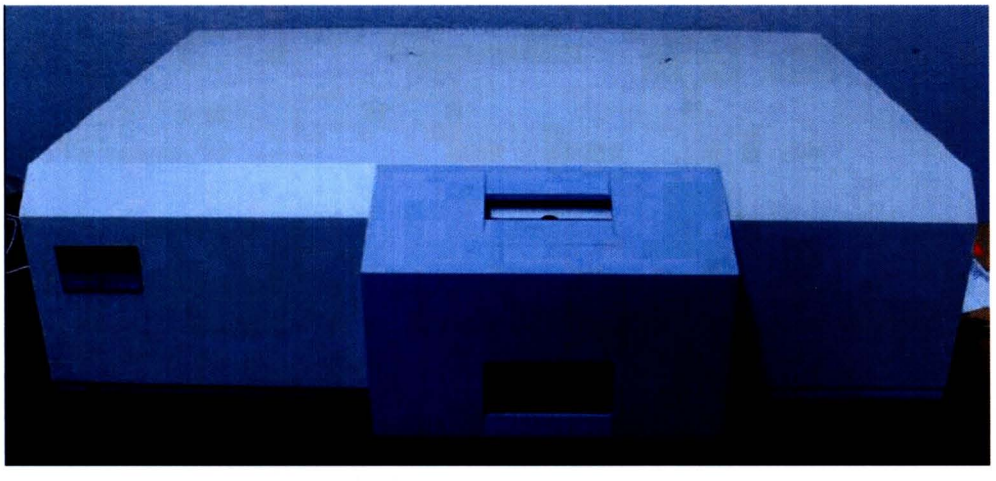

Figure 2.8 Luminescence spectroscopy

2.3.7 UV–visible Spectroscopy

UV–visible properties of products were investigated by a Lambda 25 spectrometer using UV lamp with the resolution of 1 nm.

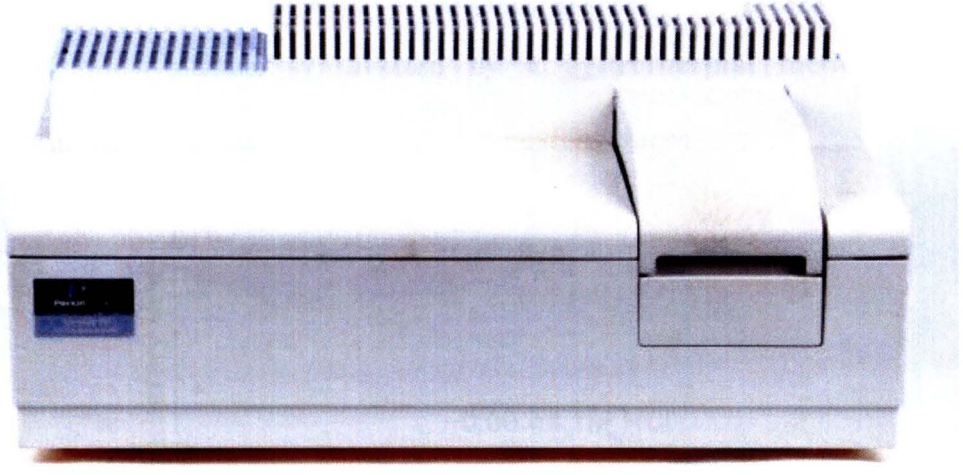

Figure 2.9 UV-visible spectroscopy