The city of Cubatão is a heavily industrialized area, located approximately 50 km from São Paulo. The city is located on a coastal basin at the base of a range of mountains that rise from 700 to 1000m, which limits air circulation, trapping air pollutants, favoring air pollution episodes. Internationally known as “the Valley of Death”, the worst polluted city in the 1980’s, Inhalable Particles annual average decreased from 168 to 90 µg.m-3 in 1990. This decrease was due to one of the biggest anti-pollution program ever mounted in Brazil. Despite this improvement in air quality, violation of PM10 24-hours standard still occurs. This study aimed at the chemical and size characterization of particulate matter from Cubatão. Throughout 2009, 5 sampling campaings were carried out at CEPEMA (Centro de Capacitação e Pesquisa em Meio Ambiente da USP), in the vicinity of PETROBRAS oil refinery. Mini-vol portable air sampler (MV) was deployed to collect coarse and fine particles. Size-fractionated particle samples were collected by a Micro-Orifice Uniform Deposition Impactor (MOUDI) device. Gravimetric analysis showed three peaks for mass size distributions: the After-Filter stage (cut point diameter < 0,1µm), stages 7A (d=0,32µm) and 3A (d= 3,2µm). Fine particle matter (FPM) concentrations were almost always lower than coarse particle matter (CPM) concentrations. Comparison between the PM$_{2.5}$ measurements by the MOUDI and MV reveals good agreement. Reflectance analysis showed that almost all the Black Carbon is found in the MV FPM and lower stages of the MOUDI, with higher concentrations at the AF.