Abstract — The optimum working conditions of a Parr laboratory autoclave for minerals dissolution were studied and the results obtained were applied to the leaching with HF of a Nb and Ta mineral, from the San Luis, Argentina. The optimized variables were heating rate, liquid volume, stirring speed, inert solid addition and cooling rate. Changes in the liquid volume, stirring speed and presence of an inert solid did not produce modifications in the actual heating rate. The optimum heating programs ranged between 5 and 10°C/min and depended on the programmed final temperature. The cooling period was 25 min. The results indicated that, using the optimum working conditions, more than 90% of mineral might be dissolved at 220°C. It was also observed that the dissolution of mineral was slightly affected by the stirring speed and the particle size whereas temperature, HF concentration, solid-liquid relation and reaction time strongly influenced the extraction of elements.

Keywords — Parr autoclave, Ores, Leaching, Operating variables, Ferrocolumbite.

I. INTRODUCTION

Metal extraction by hydrometallurgy is a technology of increasing importance in the extractive metallurgy. Leaching is one of the procedures used for the extraction of metals from minerals and concentrates and also for the refinement of metals and their recovery from the leaching solutions (Sarkar, 1983; Habashi, 2005). The use of autoclaves has greatly increased in the hydrometallurgical activities since, as they are closed vessels, they cause minor pollution (Habashi, 1971, 1999; Dreisinger, 1995). From the industrial point of view, closed vessels are more useful, when compared with other reactors, because many industrial dissolution processes usually require temperatures above the water boiling point in order to proceed at a reasonable speed (Sarkar, 1983; Dreisinger, 1995).

In the past decades, common minerals such as sulphurs (Cu, Zn, Pb, Co and Au) were treated by pressure leaching (Berezowsky, 1991). Serova et al. (1993), using an autoclave, studied the distribution of In between the aqueous and the solid phases during the oxidizing leaching of zinc sulphide concentrates. This device was also used to treat a mineral containing rare earths (Silva and Barbosa, 1993).

In the last years, many researchers have tried to determine the kinetics of mineral leaching in closed vessels. Antonijevic et al. (2004) investigated the kinetics of chalcopyrite leaching with hydrogen peroxide in sulphuric acid. Jiang et al. (2004) using the same leaching medium, studied the kinetics of pyrolusite leaching from manganese-silver associated ores. The kinetics of sand leaching in alkaline medium and of malachite in sulphuric medium was investigated by Mgaidi et al. (2004) and Bingöl and Canbazoğlu (2004), respectively.

The industrial process used to extract Nb and Ta from minerals by hydrometallurgy at atmospheric pressure makes use of HF at 70% v/v in order to obtain high dissolutions of the mineral (Gupta and Suri, 1994). Although the pressure leaching with low HF concentrations has some advantages, such as minor acid consumption, minor corrosion of the reactor, minor negative effects on the environment and fewer difficulties in the dissolution of these slight soluble minerals, there is little information about the dissolution in an autoclave of minerals containing Nb and Ta. Habashi and Toromanoff (1983) studied the dissolution of pyrochlore with HCl in an autoclave with the aim of obtaining Nb₂O₅ of technical grade. Krasilshckik et al. (1991) used pressure leaching for low solubility materials, such as Nb and Ta oxides. Rodríguez et al. (2004, 2007) have investigated the influence of carboxilic acids on the leaching of ferrocolumbite with HF and have proposed a kinetic model for the leaching of ferrocolumbite with HF.

On the other hand, a reliable kinetic study of a solid-liquid hydrometallurgical reaction in an autoclave requires a carefully determination of the equipment working conditions to obtain an adequate reproducibility and the control of the important parameters. It is important to emphasize that the optimum working conditions of an autoclave may vary from vessel to vessel. Consequently, it is essential to know them all, and the most convenient way to determine these conditions is through experimentation.

The purpose of this work is to establish the optimum working conditions of a Parr laboratory autoclave and to carry out experimental assays in order to obtain reliable data for the dissolution reaction of a ferrocolumbite with HF, under high pressure conditions.

II. MATERIALS and METHODS

A. Equipment

The equipment used was a 4562 Parr autoclave, which is shown in Fig. 1.