Presence of chemical contaminants in alcoholic beverages has become an important public health problem, mainly in Brazil, where the distillates consumption has grown year-after-year. Handmade production, without control tools of process, favors the formation of undesirable compounds, like ethyl carbamate (EC). Known to be carcinogenic, this substance is formed during the fermentation of foods and has been found in many types of yeast-distilled beverages such as sugar cane spirits, brandies, whiskies and sakes. Tiquira is a cassava spirit, typical beverage of Maranhão, Brazil. Widely consumed in the state, this product has drawn attention of researchers by high levels of EC. For this reason, this study aimed to determine the ethyl carbamate concentration by gas chromatography with mass spectrometry method (GC-MS) in 11 samples of tiquira from Maranhão. The results found varied between 26.74 and 1460.50 µg/L for this compound, with an average content of 516.94µg/L. 54.5% of the samples contained EC above the maximum allowed by Brazilian legislation for sugar cane and other spirits, equivalent to 150µg/L. The results indicate the need for changes in process conditions in order to reduce the ethyl carbamate levels in these products and to promote consumer safety.