

A simple method has been developed to determine amino acid loading in solid phase peptide synthesis. The method is applicable for the most common type of synthesis, which use Fmoc as protective group and piperidine as a deprotecting agent. Both products of deprotection reaction are separated by HPLC and determined using an UV detector; an internal standard is added. The method gives true values that have been verified by an independent method. The *RSD* is 1.52%. The method is more accurate than the published methods and allows the determination from the waste of synthesis. The employing of the internal standard eliminated the necessity of dilution accuracy or known volumes.