A series of 3-mercapto-4H-1,2,4,-triazole compounds namely 3-alkyl/aralkyl thio-4,5-disubtituted-4H-1,2,4-triazoles 1-6a,b were prepared. Their thermal decomposition pattern gave two peaks. The first one is due to their melting point followed immediately by an exothermic peak. It was found that substitution at position 3 or 5 with an alkyl group increases the thermal stability relative to compounds where a phenyl or benzyl group is substituted at these two positions. A definite correlation between thermal stability and antimicrobial activity cannot be verified in this series of compounds.