THESIS APPROVAL

GRADUATE SCHOOL, KASETSART UNIVERSITY

Master of Science (Chemistry)

DEGREE

Chemistry	Chemistry
FIELD	**DEPARTMENT**

TITLE: A Coupling of Superheated Water and Solid-Phase Extraction of *N*-nitrosamine from Frankfurters

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THESIS

A COUPLING OF SUPERHEATED WATER AND SOLID-PHASE EXTRACTION OF *N*-NITROSAMINE FROM FRANKFURTERS

PANEE SUBPRASERT

A Thesis Submitted in Partial Fulfillment of
the Requirements for the Degree of
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Panee Subprasert 2007: A Coupling of Superheated Water and Solid-Phase Extraction of *N*-nitrosamine from Frankfurters. Master of Science (Chemistry), Major Field: Chemistry, Department of Chemistry. Thesis Advisor: Assistant Professor Orapin Chienthavorn, Ph.D. 125 pages.

An efficient, rapid and inexpensive method for extraction and clean-up of selected volatile nitrosamines, namely nitrosodiethylamine (NDEA), nitrosopiperidine (NPIP), nitrosopyrrolidine (NPYR) and nitrosomorpholine (NMOR) from frankfurters was developed by using superheated water (SW) coupled with solid-phase extraction (SPE). A number of parameters affecting SPE, such as sorbent types, eluting solvent system and its volume, were optimized to obtain the highest yield. Since a coextraction of lipid caused serious problem during the extraction, a subsequent clean-up step using fat-selective florisil adsorbent with 60% ethyl ether-dichloromethane was then necessary. Likewise, various factors affecting superheated water extraction (SWE), such as flow rate, extraction temperature, dynamic time and static time were also investigated. A full factorial design with three replicates was employed to study an influence of several parameters on SWE in terms of recovery. The extraction temperature was optimized at 140 ^{o}C with dynamic and static time of 10 and 5 min, respectively, at a flow rate of 1 mL min^{-1}. Separation and quantification of nitrosamines were carried out using GC-FID and GC–MS in selected ion monitoring (SIM) mode. Direct application of this method to frankfurter samples allowed overall recoveries of *N*-nitrosamines to be in a range of 81-106 % with a relative standard deviation less than 10 %. The detection limit of the coupling method ranged from 0.47 to 1.48 ng nitrosamine injected. Principal advantages of this method are time saving, low consumption of organic solvent, together with a few minor advantages of the simplicity of instrument and a coupling of extraction and clean-up step.

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Student's signature Thesis Advisor's signature

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