

isomers that may be present as well as related compds., such as piperidine and piperidine, that also absorb at 340–345 nm.

B. Apparatus and Reagents

(a) *UV spectrophotometer*.—Beckman A-25 (replacement instrument model DU-641), or equiv., with 1 cm sq. silica cuvetts.

(b) *Volumetric flasks*.—100 mL g-x, amber (No. 161-9057, Luxco Scientific). Amber flask is required to reduce photo-degradation of piperine in soln.

(c) *Ethylene dichloride*.—Reagent grade 1,2-dichloroethane (J.T. Baker, Inc., No. H0763). Make all dilns with this solv.

(d) *Piperine*.—Aldrich Chemical Co., No. P4,900-7.

C. Determination of Absorbance Factor

Weigh 0.1000 g piperine into 100 mL vol. flask, add ca 70 mL $C_2H_2Cl_2$, shake to dissolve, and dil. to vol. Pipet 10 mL into 100 mL vol. flask and dil. to vol. Into six 100 mL vol. flasks, pipet 1, 2, 3, 4, 5, and 6 mL aliquots, resp., and dil. each to vol. Zero spectrophot. with $C_2H_2Cl_2$, and read *A* of each final diln at max. 342–345 nm using UV light source and $C_2H_2Cl_2$ in ref. cell. Divide each reading by mL used in final diln to convert *A* to concns of $1/10^3$ (µg./L.). Det. av. of 6 values. Reciprocal of this av. is concn, mg/mL, of piperine required for *A* of 1.000, and is factor (*F*) used for calcs.

D. Procedure

(a) *Black and white pepper*.—Grind and screen sample to pass 30 mesh NIST sieve and blend uniformly. Accurately weigh (± 0.0001 g) 0.5 g sample and transfer to 125 mL erlenmeyer. Protect from light. Add ca 70 mL $C_2H_2Cl_2$. Reflux and stir 1 h, cool to room temp., and filter quant. thru paper into 100 mL vol. flask. Transfer rest of exd residue to filter, wash thoroly, and dil. to vol. Pipet 2 mL of this soln into 100 mL vol. flask and dil. to vol. Record *A* at max. 342–345 nm.

(b) *Pepper oleoresins*.—Accurately weigh (± 0.0001 g) 1.0 g well mixed sample and transfer to 100 mL vol. flask. Dil. to vol. with $C_2H_2Cl_2$. Shake well until dissolved. Pipet 10 mL into 100 mL vol. flask and dil. to vol. Pipet 1 mL of this soln into 100 mL vol. flask and dil. to vol. Record *A* at max. 342–345 nm.

(c) *Soluble pepper seasonings*.—Accurately weigh (± 0.0001 g) 2.0 g sample and transfer to 100 mL vol. flask. Add ca 70 mL $C_2H_2Cl_2$ and let stand 20 min, swirling occasionally. Dil. to vol. and let settle. Pipet 1 mL into 100 mL vol. flask and dil. to vol. Record *A* at max. 342–345 nm.

E. Calculations

$$\% \text{ Piperine} = [(A_s \times F \times V)/(W_s \times 10^3)] \times 100$$

where *A_s* = absorbance of sample; *F* = factor derived from piperine std; *V* = diln vol., mL; *W_s* = sample wt., g.

Ref.: JAOAC 70, 112 (1987).

CAS-94-62-2 (piperine).

920.170 Prepared Mustard Preparation of Sample Procedure

Transfer entire contents of container to dish large enough to permit thoro stirring and make whole mass homogeneous. Preserve in g-x bottle. Stir well each time before removing portion for analysis.

920.171 Solids in Prepared Mustard Gravimetric Method Final Action

Weigh 5 g sample into flat-bottom Pt dish with approximately 5 g dry sand; distribute evenly over bottom of dish with little H_2O , place on steam bath until moist. appears dry, and heat in oven at 100° to min. wt.

941.13 Chlorides (Total) in Prepared Mustard Titrimetric Method Final Action

Weigh 3–4 g sample from weighing bottle, place in 300 mL erlenmeyer, and add excess std 0.1*N* $AgNO_3$ (usually 30 mL is enough). Mix thoroly, add 15 mL HNO_3 , and bring to boil on hot plate. To boiling mixt. add 15 mL 5% $KMnO_4$ soln, 5 mL at time, rotating flask after each addn to mix contents. Add ca 50 mL H_2O and filter into 200 mL vol. flask. Wash filter free of $AgNO_3$ and dil. to vol. with H_2O . Mix thoroly and titer 100 mL aliquot with 0.1*N* $KSCN$, using 2 mL stdd Fe alum soln as indicator. Calc. chlorides as NaCl.

Refs.: JAOAC 24, 703 (1941); 25, 97 (1942).

CAS-7647-14-5 (sodium chloride)

920.172 Ether Extract of Prepared Mustard Gravimetric Method Final Action

(*Caution*: See safety notes on monitoring equipment, distillation, flammable solvents, and diethyl ether.)

Weigh 10 g sample into SiO_2 , Al, or porcelain drying dish and mix with ca 30 g sand. Heat on H_2O bath until moist. appears dry; then finish drying in oven at 100°. Grind until all lumps are broken up, and det. ether ext by extg 16 hr with anhyd. ether in Soxhlet extractor with Whatman single thickness or other close-texture thimble. Dry ext 30 min at 100°, cool, and weigh.

920.173 Nitrogen (Total) in Prepared Mustard Improved Kjeldahl Method Final Action

Det. N as in 955.04C, using 5 g sample.

920.174 Acidity of Prepared Mustard Titrimetric Method Final Action

Weigh 10 g sample into 200 mL vol. flask, dil. to vol. with H_2O , shake, filter thru dry paper, and det. acidity in 100 mL by titer with 0.1*N* alkali, using phenol. Express result as HClAc. 1 mL 0.1*N* alkali = 0.0060 g HClAc.

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A Fajgelj, A Ambrus



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Handbook of Food Analytical Chemistry, Volume 1 Ronald E. Wrolstad, Terry E. Acree, Eric A. Decker, Michael H. Penner, David S. Reid, Steven J. Schwartz, Charles F. Shoemaker, Denise M. Smith, Peter Sporns, 2005-09-16 Emphasizing effective state of the art methodology and written by recognized experts in the field the Handbook of Food Analytical Chemistry is an indispensable reference for food scientists and technologists to enable successful analysis Provides detailed reports on experimental procedures Includes sections on background theory and troubleshooting Emphasizes effective state of the art methodology written by recognized experts in the field Includes detailed instructions with annotated advisory comments key references with annotation time considerations and anticipated results *Handbook of Toxicology of Chemical Warfare Agents* Ramesh C Gupta, 2015-01-21 Handbook of Toxicology of Chemical Warfare Agents Second Edition covers every aspect of deadly toxic chemicals used in conflicts warfare and terrorism Including findings from experimental as well as clinical studies this essential reference offers in depth coverage of individual toxicants target organ toxicity major incidents toxic effects in humans animals and wildlife biosensors and biomarkers on site and laboratory analytical methods decontamination and detoxification procedures and countermeasures Expanding on the ground breaking first edition Handbook of Toxicology of Chemical Warfare Agents has been completely updated presenting the most recent advances in field Brand new chapters include a case study of the Iran Iraq war an overview of chemical weapons of mass destruction explosives Ricin the human respiratory system alternative testing methods brain injuries and more Unites world leading experts to bring you cutting edge agent specific information on Chemical Warfare Agents CWA and their adverse effects on human and animal health and the environment Provides you with all the information you need on CWA modes of action detection prevention therapeutic treatment and countermeasures New to this edition a full update to reflect the most recent advances in the field and new chapters on emergency preparedness the chemical warfare agents used in Syria and the use of the Novichok agent in the UK *Principles and Practices of Method Validation* A Fajgelj, A Ambrus, 2007-10-31 Principles and Practices of Method Validation is an overview of the most recent approaches used for method validation in cases when a large number of analytes are determined from a single aliquot and where a large number of samples are to be analysed Much of the content relates to the validation of new methods for pesticide residue analysis in foodstuffs and water but the principles can be applied to other similar fields of analysis Different chromatographic methods are discussed including estimation of various effects eg matrix induced effects and the influence of the equipment set up The methods used for routine purposes and the validation of analytical data in the research and development environment are documented The legislation covering the EU Guidance on residue analytical methods an extensive review of the existing in house method validation documentation and guidelines for single laboratory validation of analytical methods for trace level concentrations of organic chemicals are also included With contributions from experts in the field any practising analyst dealing with

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and the advantages of using suitable reference materials Finally the benefits to both the analytical laboratory and the user of the results are considered

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Chemistry International, 1995

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