Summaries of Official Spectrophotometric Methods

1) Drug analysis

Introduction

Standard methods for routine testing of materials are published by the Association of Official Analytical Chemists (AOAC). AOAC methods are reliable because they have been collaboratively tested by a minimum of 8 labs and evaluated by authorities in the particular field.

They are jointly developed by government, industry and university. Subsequently the methods are continuously tested and updated. Laboratories in various areas of commerce rely on the AOAC as an authoritative source because benefits for users have been gained in various ways, including:

- saving time and money in performing unreliable methods
- satisfaction of quality assurance program recommendations by using validated methods
- acceptance by regulatory authorities and courts
- recognition world-wide for trade

These methods are listed with wavelengths providing quick reference to the spectrophotometer requirements. Spectrophotometers and Acquire software from the Biochrom Libra range are recommended for each method as appropriate. More details on instrument settings are shown in the user manuals and other application notes.

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Summary of Analytical Methods - Drug Analysis (Libra Application Note 49)

COMMODITY ANALYSIS	OUTLINE OF METHOD	WAVELENGTHS nm	RECOMMENDED INSTRUMENT					
			LIBRA S5	LIBRA S12	LIBRA S22	LIBRA S22 + ACQUIR	LIBRA S32	LIBRA S32PC
Acetaminophen and salicylamide in drugs	Ads column extract Etac. Etac/CHCl3	210-320, (248). 260-370. (308)				x	x	x
Acetaminophen in drugs	Diatom earth column, extract with ether	240-350, read 249				x	x	x
Alkaloids (total)	Ethanol extract	250-260		x	x		x	x
Amphetamine drugs	Extract with CHCl3, NaOH.	252-280 max, min				x	x	x
Antihistamines in drugs with aspirin phenacetin and caffeine	Acidic solution from CHCl3 extract	264, 265 and 314		x	x		x	x
Arsenic and phenobarbital in drugs	CHCl3 extr, ads col	Phenobarbital 240.5,aspirin 278				x	x	x
Bendroflumethiazide in drugs	Sod carbonate, silicate column extract	271		x	x		x	x
Benzocaine and antipyrene in drugs	Column partition chromatography	283		x	x		x	x
Benztropine mesylate in drugs	Acid extr with bromomphenol blue	410	x	x	x		x	x
Butabarbital sodium in drugs	Extract in aqueous DMSO.	Scan max 239				x	x	x
Chloral hydrate in drugs	Quinaldine/ethyl iodide forms cyanine dye.	605	x	x	x		x	x
Codeine and terpin hydrate in elixirs	Separate extracts aq. ethanol/ptoluenesulphonic acid.	287. Terpin+phosphotungstic acid 725.		x	x		x	x
Dichlorophene in drugs	CHCl3 extr. NaOH soln	305		x	x		x	x
Dienestrol in drugs	Alumina column purifcation	240-400, max 303				x	x	x
Diethylstilbestrol in drugs	CHCl3 extr, ammonia+sod nitrite convert to phenols	420	x	x	x		x	x
Digitoxin in drugs	Silica column/CHCl3, alk picrate reagent	495	x	x	x		x	x
Digoxin and total digitoxosides in drugs	Alkaline dinitrobenene	596 + 620	x	x	x		x	x
Ephedrine in solid dosage drugs	Basic column+CHCl2 elution. Periodate reaction	230-320 (benzaldehyde)				x	x	x
Ethinyl estradiol in drugs	Adsorption column/CHCl3 extr.	500-700. 537 max				x	x	x
Hexestrol in drugs	Ethanol extract	Scan 215-320				x	x	x
Iron in drugs	Acidic solution. Complex from 1:1dipyridyl.	Scan 500-700. max 525.				x	x	x
Mannitol hexanitrate(1) and phenobarbitol(2) in drugs	(1) React phenoldisulphonic acid (2) Acetate extr.	240 & 408		x	x		x	x
Menadione sodium bisulfite in drugs	Silica column	280-400		x	x		x	x
Mephentermine sulfate in drugs	Ion exchange column. Ethanol/HCl extract	254-262		x	x		x	x
Mestranol in drugs	Silica column/hexane extract.	287-302-315 with baseline subtraction				x	x	x
Mestranol with ethynodiol diacetate in drugs	Partition column. Methanol/sulphuric acid extract	540	x	x	x		x	x

Methyl salicylate in drugs	CHCl3 extract.	305		x	x		x	x
Neostigmine methylsulfate in drugs	Alkaline hydrolysis	230-350		x	x		x	x
Niacinamide in multivitamin preparations	KH2PO4 extract/pH 4.5/CNBr+barbituric acid	550	x	x	x		x	x
Norepinephrine in epinephrine preparations	Acid extract/acetylacetone+adsorption column	520	x	x	x		x	x
P-aminosalicylic acid and isoniazid in drugs	Extract with sod bicarbonate	299,244,325. CHCl3 extract,302, 375				x	x	x
Papain proteolytic activity	Casein hydrolysate/protein precipitate.	280		x	x		x	x
Pentaerythrityl tetranitrate in drugs	Phenoldisulphonic acid reagent	408	x	x	x		x	x
Phenobarbital and aminophylline/theophylline in drugs	1) Ether/HCl extract. 2) Aqueous residue	1) 240.5 2)271		x	x		x	x
Phenobarbital and phenytoin in drugs	CHCl3/amyl alcohol extract. Adsorption column	1) 253 2)258-263		x	x		x	x
Phenobarbital and theobromine in drugs	CHCl3/sulphuric acid extract.	1) 240.5 2)274		x	x		x	x
Phenylethlamines in drugs	0.1N sulphuric acid + CHCl3	252-263		x	x		x	x
Phenylpropanolamine hcl in drugs	Alumina column + CHCl3	258.5		x	x		x	x
Phenytoin sodium in drug capsules	DMSO/HCl + Silica column. CHCl3 extract.	258		x	x		x	x
Polythiazide in drugs	DMSO extract. Silica column isooctane/ether elution	268		x	x		x	x
Procainamide hcl in drugs	Acid extract + CHCl3. Alkaline solution	272		x	x		x	x
Progestational steroids in drugs	CHCl3 extract. Isonicotinic acid hydrazide reaction.	380	x	x	x		x	x
Propylthiouracil in drugs	Dissolve in dilute ammonia	234		x	x		x	x
Rescinnamine in drugs	Ethanol + CHCl3 extract. NaNO2/amm.sulphamate	390	x	x	x		x	x
Reserpine-rescinnamine alkaloids in Rauwolfia serpentina	Ethanol extract. NaNO2/amm.sulphamate/H2SO4	390	x	x	x		x	x
Reserpine in drugs	Ethanol extract. NaNO2/amm.sulphamate/H2SO4	390	x	x	x		x	x
Rutin in drugs	Acid/ethanol extract	338.5, 352.5, 366.5				x	x	x
Thiazide drugs:Benz,chlor,hydrochor&hydrofl,methycl.	Silica column elution	Benz295,chl278,hyd273 met268				x	x	x
Trisulfapyrimidines in drugs	Couple with N naphthylethylenediamine	480-660	x	x	x		x	x