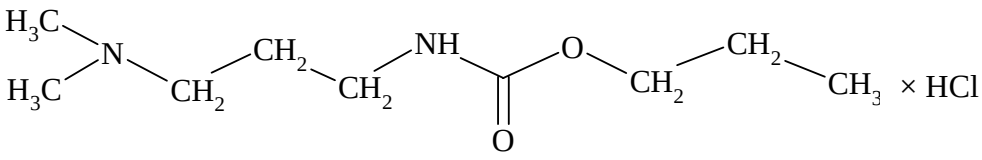
PROPAMOCARB HYDROCHLORIDE
399



ISO common name	Propamocarb-hydrochloride
Chemical name	Propyl 3-(dimethylamino) propylcarbamate hydrochloride (IUPAC, CA; *25606-73-5*) propyl 3-(dimethylamino)propylcarbamate (propamocarb CA; *24579-73-5*)
Empirical formula	$C_9H_{21}ClN_2O_2$ ($C_9H_{20}N_2O_2$ propamocarb)
RMM	224.73 (188.27 propamocarb)
m.p.	55°C
v.p.	0.8 Pa at 25°C
Solubility	At 25°C in water and methanol: 700 g/l; dichloromethane: 430 g/l; ethyl acetate: 23 g/l; hexane and toluene: 100 mg/l
Description	Propamocarb hydrochloride is a colourless, crystalline, hygroscopic solid
Formulation	Aqueous solution

PROPAMOCARB-HYDROCHLORIDE AQUEOUS SOLUTION
[*]**399**/SL/M/-

1 Sampling. Take at least 250 ml.

2 Identity tests

2.1 Infrared. Apply a thin film of sample between calcium fluoride discs. Scan the spectrum between 4000 to 400 cm^{-1}. The spectrum produced from the sample should not differ significantly from that from an authentic standard solution produced in the same way.

[*] CIPAC method 1991. Prepared by the German Committee (DAPA) Chairman: Dr W Dobrat. Based on a method supplied by Schering (FRG)

2.2 HPLC. Use the HPLC method below. The relative retention volumes of the sample should not deviate by more than 1% from that for the calibration solution.

3 Propamocarb hydrochloride

OUTLINE OF METHOD The sample is dissolved in methanol and separated by liquid chromatography on a silica column. The content of propamocarb hydrochloride is determined from peak areas using external standard calibration.

REAGENTS

Propamocarb hydrochloride reference standard, aqueous concentrate with a guaranteed content of propamocarb hydrochloride
Water HPLC grade
Methanol HPLC grade
Ammonia solution 25% w/w
Solvent mixture methanol-water: 80 + 20 (v/v)
Eluent. Dilute 8 ml of the ammonia solution with 192 ml water and 800 ml methanol.
Calibration solution. Weigh (to the nearest 0.1 mg) about 175 mg (*s* mg) of propamocarb hydrochloride into a volumetric flask (100 ml) and fill up to the mark with solvent mixture.

APPARATUS

Liquid chromatograph preferably with a pump with rinsing heads, equipped with an UV spectrophotometer (e.g Knauer Model 8700)
Column stainless steel, length 250 mm × 4,6 mm (i.d.), packed with LiChrosorb Si 100, 10 µm

PROCEDURE

(a) Operating conditions (typical)
Flow rate 1.0 ml/min
Detector wavelength 210 nm
Temperature ambient
Injection volume 10 µl
Chart speed 0.5 cm/min
Capacity factor 4.17 for propamocarb hydrochloride

(b) preparation of sample. Weigh (to the nearest 0.1 mg) into a volumetric flask (100 ml) sufficient sample to contain about 200 mg propamocarb hydrochloride (*w* mg) and fill to the mark with solvent mixture.

(c) Calibration. Inject 10 µl portions of the calibration solution into the chromatograph until the peak areas of successive injections differ by less than 1%. Otherwise repeat the calibration.

(d) Determination. inject in duplicate 10 µl portions of the sample solution followed by an injection of the calibration solution. Measure the peak areas. Calculate the mean of the areas of the peaks of the calibration injections preceding and following the sample injections and use this value for the calculation of the two bracketed sample runs.

(e) Calculation. When using a suitably programmed data system, the content of propamocarb-HCl will be directly printed out. It is calculated from the following formula:

$$\text{Propamocarb hydrochloride content} = \frac{H_w \times s \times P}{H_s \times w} \text{ g/kg}$$

where:

s = mass of propamocarb-HCl (reference standard) in calibration solution (mg)
w = mass of sample taken (mg)
H_w = peak area of propamocarb-HCl in the sample solution
P = purity of the reference standard (g/kg)

Repeatability r = 3.8 to 12.4 g/kg at 640 to 655 g/kg active ingredient content
Reproducibility R = 6.9 to 25.5 g/kg at 640 to 655 g/kg active ingredient content