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Spectrophotometric Determination of Novalgin in Pharmaceutical Preparations by the Molybdenum Blue Method

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A colour reaction has been developed for the determination of novalgin in pharmaceutical preparations. The method is simple and sensitive with molar absorptivity 3.8 x 10^4l $mole^{-1}$ cm^{-1}. Novalgin is determined Spectrophotometrically by molybdenum blue method. Beer's law is obeyed in the concentration range of 1-10 µg/ml of novalgin.

NOVALGIN (Analgin or dipyrone) is the sodium salt of [(2,3-dihydro-1, 5-dimethyl -3-oxo-2-Phenyl-1 H-Pyrazol-4-yl) methylamino] methane Sulphonic acid. It is a commonly used analgesic drug. Its determination in pharmaceutical preparations is, therefore important. An indirect spectrophotometric method for the determination of novalgin in tablets by use of potassium iodate has been developed.[1] Another spectrophotometric method for the determination of novalgin has been studied by Qureshi and coworkers.[2] The method is based on reduction of iron (III) with novalgin and subsequent complexation of iron (II) with 1,10-phenanthroline. N-bromosuccinimide in acetic acid medium has been used as an analytical reagent for spectrophotometric determination.[3] In another reaction hydrolysed novalgin reacts with phenol and potassium ferrocyanide giving an orange red colour[4] that absorbs maximally at 525 nm. Buhl and Hachula[5] have reported an indirect spectrophotometric method based on reduction of Ce (IV) to Ce(III) by novalgin and its subsequent determination with arsenazo III. Similarly in few other methods the reducing interaction of a number compounds, such as tetracycline, cystein, and ascorbic acid[6-8] have been utilized for their determination. The methods are based on their interaction with ammonium molybdate and phosphoric acid to produce the blue colour which is regarded as molybdenum blue. In our studies, novalgin has also been found to inteact with ammonium molybdate and phosphoric acid to produce the blue colour solution. This colour reaction has been studied for spectrophotometric determination of drug.

An ECIL GS 866 D spectrophotometer (manufacturer) with 10 mm matched quarz cells was used.

All reagents used were of analytical grade. Solutions of 0.005 M ammonium molybdate and 1.0 M phosphoric acid were prepared in distilled water.

Onen hundred mg of novalgin was dissolved in 100 ml of distilled water. A 10 ml was diluted portion accurately to 100 ml with distilled water to obtain a working standard of 100 µf/ml solution for the preparation of calibration graph.

To an aliquot volume of 0.1 ml to 1.0 ml containing 10 µg/ to 100 µg of novalgin 2ml of ammonium molybdate, 2 ml of phosphoric acid and 4 ml of distilled water was added

Table 1 Results for determination of novalgin by proposed method compared with official method[9] and reference method[1]

Drug	Nominal Composition (mg)	Recovery *%		
		Proposed method	Official method	Reference method
Baralgan	500 analgin (novalgin) 5 p-piperidinoethoxy-O-carbmethoxy-benzophenone hydrochloride 0.1 diphenyl-piperidinoethyl acetamide brom-O-methylate	99.4 ± 0.7	101.0 ± 0.5	101 ± 0.3
Spasmizol	500 analgin (novalgin) 10 phenobarbitone 2.5 homatropin methyl bromide	99.6 ± 0.4	100.0 ± 0.3	100.8 ± 0.1
Maxigon	500 analgin (novalgin) 5 p-piperidinoethoxy-O-carbmethoxy-benzophenone hydrochloride. 0.1 diphenyl-piperidinoethyl acetamide brom-O-methylate 7.5 hydroxyzinen hydrochloride.	100.8 ± 0.2	103.5 ± 0.8	99 ± 0.4
Spasmolysin	500 analgin (novalgin) 10 dicyclomine hydrochloride	98.9 ± 0.7	103.0 ± 0.5	102.6 ± 0.1
Ultragin	250 analgin (novalgin) 250 paracetamol 25 caffeine	102.3 ± 0.4	102.8 ± 0.7	102.4 ± 0.6
Zimalgin	250 analgin (novalgin) 250 paracetamol 15 coffeine 5 codein phosphate	101.9 ± 0.5	103.3 ± 0.6	102 ± 0.8

*Mean ± S.D. for 5 determinations, based on label claim.

in a boiling tube. The content, was heated on boiling water bath for 20 minutes. After cooling it was transferd to a 10 ml volumetric flask. The solution was made upto the mark with distilled water and the absorbance was measured at 800 nm against reagent blank. The colourwas found to be stable for more than one hour.

An aliquot of the finely divided powder of novalgin tablet containing about 50 mg of the active principle was extracted in 30 ml distilled water and allowed to stand for 15 minutes. Subsequently filtered into a 50 ml volumetric flask through a Whatman No. 42 filter paper. A 10 ml portion was dedicted accurately to 100 ml was distilled water to obtain a final solution containing 100 µg of drug per ml. Half on ml of this solution was taken and subjected to the following procedure.

Beer's law was obeyed in the concentration range of 1-10 µg/ml of novalgin with molar absorptivity 3.8×10^4 1 mol^{-1} cm^{-1}. The determination of novalgin was done in various pharmaceutical preparation and the results are shown in Table 1. Some excipients and other drugs commonly added to dosage forms were found not to interfere. These are glucose, fructose, starch. aspirin, codein sulphate, phenylbutazone, oxyphenbutazone, diazepam, caffein, paracetamol, propylphenazone and phenacetin.

Tetracycline, cystein and ascorbic acid have been determined spectrophotometrically by the molybdenum blue method[6-8]. All these drugs are reducing agents when treated with ammonium molybdate in presence of phosphoric acid, molybdate ion is reduced to molybdenum blue. Novalgin is also a reducing substance that can reduce ammonium molybdate to molybdenum blue in presence of phosphoric acid. The recommended method presented in this communication is advantageous for the determination of novalgin in microgram range.

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