TABLE 2: ASSAY OF NTG IN TABLETS

Formulation	Label claim mg/tablet	Amount found by proposed methods		% Recovery by proposed Method*
		Method A	Method B	
Tablet 1	60	59.7±0.05	59.5±0.06	99.9±0.08
Tablet 2	120	119.9±0.08	119.8±0.05	99.9±0.92

Tablet 1 is Glinate, 60 mg manufactured by Glenmark Pharma, Mumbai and tablet 2 is Natelide, 120 mg, Alembic, Vadodara. *Recovery of 10 mg added to the pre-analyzed pharmaceutical dosage forms (average of 3 determinations).

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FTIR-Spectrum of Galactomannan Extracted from *Trigonella foenum - graceum*

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Guar is used in millions of tones annually by various industries and is employed medicinally as well. The galactomannan portion of the guar seed is found to be active and responsible for its numerous industrial and medicinal applications. Guar galactomannan has a galactose-mannose ratio (G:M) of 1:2 and possesses a very high viscosity. The objective of this study was to explore some other source of galactomannan having different G:M ratio and to study its effect on medicinal applications. Fenugreek is one such promising source that offers a galactomannan having a G:M ratio of 1:1. The extracted galactomannan from fenugreek was compared with guar galactomannan on the basis of FTIR-spectra. The peaks and their accompanying shoulders, in the FTIR-spectra of the two compounds, were found to overlap closely.

The literature survey on *Trigonella foenum-gracium* or fenugreek, reports its numerous (about 30) medicinal applications[1,2]. The mechanism and the specific constituent responsible for most of its activities are not known or simply the whole seed powder is reported to have a particular activity in question in most of the cases[3-7]. Hence, there is a need to carry out research work to correlate the active constituents of this little seed with its numerous therapeutic actions.

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The work on guar galactomannan[8-12] has extensively been carried out, while the work on galactomannans from other leguminous seeds[13-20] has been carried out to some extent. But, the work on fenugreek galactomannan is lacking. Work on other constituents of fenugreek, such as its alkaloids, glycosides, oil and saponins[21-22] has been carried out. Galactomannans are found as reserve food material in the endosperms of leguminous plant seeds. These are referred to as galactomannans, because they consist of β-(1-4)-mannose backbone having single α-(1-6)-galactose side chains[23]. The present study was taken up with a view to extract fenugreek galactomannan and to compare its FTIR spectrum with guar galactomannan.

The fenugreek seeds were ground mechanically using a mortar and pestle. The coarse sized seed powder (100g) was moistened with petroleum ether (60-80), packed in a Soxhelet apparatus and extracted with petroleum ether (60-80). Further, the extraction was carried out using absolute alcohol. Finally, the material from the extraction chamber was collected and macerated with 1.3 l of hot water (50-55°). The mucilaginous solution was filtered. Alcohol was added to the filtrate to precipitate the galactomannan. The solution was filtered and the precipitate of galactomannan was collected on muslin. The collected galactomannan was lastly washed with acetone and dried. The dried gummy mass was ground to fine powder. The yield of fenugreek galactomannan was found to be 10 % w/w.

The product obtained was water dispersible, which gets precipitated by the addition of alcohol and was free of proteins and starch. This is the minimum requirement and definition of galactomannans as per the Association of Official Analytical Chemists (AOAC). Further, to establish the identity of the extracted compound as galactomannan, acid hydrolysis, chromatographic studies and qualitative tests for absence of other constituents like alkaloids, saponins, proteins and starch were performed. Its viscosity, molecular weight and FTIR spectrum were also determined in comparison with the reference standard guar gum. In the present manuscript, the FTIR-spectrum of the extracted fenugreek galactomannan is being reported.

The FTIR-spectrum was recorded on a 8300 Shimadzu FTIR in potassium bromide (anhydrous IR Grade) pellets. Similarly, the spectrum of guar gum (gifted by Sunita Mine Chemicals, Jodhpur) was recorded. The overlain spectra of both the compounds are shown in fig. 1.

Since, the compounds being analyzed are galactomannans, having predominance of -OH groups, it is expected that these shall show absorption peaks in the region of 3200-3700 cm^{-1}, which is actually the case. In the finger print region, peaks at 407, 1020, 1080, and 1175 cm^{-1} for the two compounds are similar. The other matching peaks are at 1625 cm^{-1} and 2860 cm^{-1} accompanied with the characteristic shoulder, as can be seen in fig. 1. Based on the similar spectra recorded for the two galactomannans, fenugreek galactomannan can be expected to be pure and having similar galactomannan content as guar.

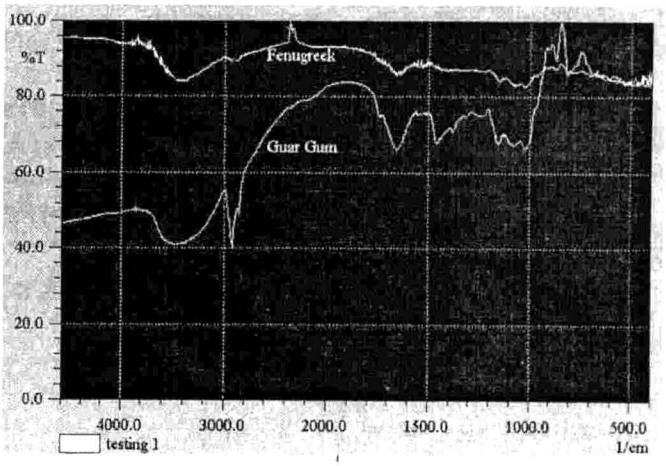

Fig.1: Overlain spectra of fenugreek galactomannan and guar galactomannan.

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Spectrophotometric Estimation of Valdecoxib in Pure Form and Tablets

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Simple UV and third derivative spectrophotometric methods have been developed for the determination of valdecoxib in bulk drug and its tablets. In simple UV spectrum of valdecoxib in 0.1 N sodium hydroxide, it exhibits absorption maxima (λ_{max}) at 243 nm where as in third derivative spectrum it shows maxima at 221.2 nm and minima at 213.6 nm. Both the methods were found to be simple, economical, accurate, reproducible and can be adopted in routine analysis of valdecoxib in bulk drug and its tablets.

Valdecoxib (VXB) is a new NSAID, which chemically is 4-[5-mehtyl–3-phenylisoxazole-4-yl] benzenesulfonamide. It is active at low dose and has less gastric toxicity. It inhibits synthesis of prostaglandins by inhibiting the activity of the enzyme, cyclooxygenase-2 (COX-2) [1-4]. It is 28,000 times more selective for COX-2 than COX-1[5]. Valdecoxib is preferred over conventional NSAIDs as they may lead to serious gastrointestinal complications such as ulcer, severe bleeding and perforation, resulting in hospitalization and even death[6-7]. It is mainly used for the osteoarthritis, rheumatoid arthritis and dysmenorrhea[8-10]. The drug is available in tablet form and is not yet official in any pharmacopoeia.

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So far only solid-phase extraction–liquid chromatography–tandem mass spectrometry method has been reported for the estimation of valdecoxib[11]. But this method is comparatively time consuming and expensive when compared to simple spectrophotometric method. The aim of the present investigations is to develop a simpler, rapid and cost-effective analytical method for the determinations of valdecoxib in bulk drug and in its various dosage forms. The present investigation illustrates two simple, sensitive and accurate simple UV method and third derivative spectroscopic method for the analysis of valdecoxib in bulk drug and in tablets.

A Shimadzu UV–1601PC UV/Vis spectrophotometer was used for all absorbance measurement. Valdecoxib was obtained as a gift sample from Cadila Pharma Ltd., Ahmedabad. Stock solution of valdecoxib (1 mg/ml) was pre-