GC-MS AS A TOOL FOR CARBOHYDRATE ANALYSIS IN A RESEARCH ENVIRNOMENT

By

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Abstract

GAS chromatography, coupled with mass spectrometry (GC-MS) as a detection technique, is a powerful analysis tool in the hands of the research chemist. The fundamentals of the technique and its application to the analysis of carbohydrates using volatile derivatives such as trimethylsilyl ethers are described. Mass fragmentation spectra are shown to be useful in indicating carbohydrate ring structure (pyranoside vs furanoside), the reducing nature of the saccharide and the type of some glycoside linkages. The use of methylation and hydrolysis (typically called methanolysis), followed by silylation and GC-MS analysis, is shown to be a useful technique to characterise oligomer and polymeric carbohydrate material (gums and films). The use of these techniques using model compounds and use in one of the Sugar Milling Research Institute's research programs will be discussed.

Introduction

The new Research Strategy of the Sugar Milling Research Institute (SMRI) has resulted in the creation of SMRI Strategic Research Thrust Areas and the move toward a medium-to-long term research focus (Dewar and Davis, 2007). To enable this shift in focus and achieve the required outcomes requires a commitment to both human and physical resources. As part of this resourcing, the SMRI has invested in new equipment including a gas chromatograph coupled to a mass spectrometer (GC-MS). This is an established analytical tool for obtaining both compound separation within a mixture and structural information on the separated organic compounds. It is used routinely in environmental analysis, forensics (profiling for drugs of abuse), arson analysis, food contamination and for identification and characterisation in the development of new pharmaceuticals.

The coupling of a mass spectrometer as a detector to a gas chromatograph was developed during the 1950s and 1960s (Gohlke and McLafferty, 1993). However, the type and size of the mass spectrometers available precluded their use in routine analysis. It was not until the 1980s, with the advent of the personal computer, that 'benchtop' instruments became available and the routine use of GC-MS became commonplace.

Since carbohydrates are thermally unstable, GC is an unsuitable method of analysis, unless the carbohydrate can be modified to make it volatile. The preparation of volatile derivatives such as methyl ethers, acetates and trimethylsilyl (TMSi) ethers was shown to be amenable to GC analysis (McInnes *et al.*, 1958; Gunner *et al*, 1961; Sweeley *et al*, 1963).

This development was followed by the use of GC-MS in carbohydrate structural and identification research (DeJongh *et al.*, 1969). Advances in GC-MS equipment and columns have resulted in the method becoming useful in compositional and structural analysis of monosaccharides, oligomers and polymers, especially in the environmental and life sciences fields.

Examples of the diverse use of the method include differentiation of strains of *Streptococcus pneumoniae* based on the capsular polysaccharide (Kim *et al*., 2005), the identification and quantification of chitin in support of dating in the fossil record (Flannery *et al*., 2001) and the analysis of plant gums used in works of art to check for forgeries (Bonaduce, 2007). This paper serves as an introduction to the technique and its use within a sugar research environment.

Experimental

Samples were prepared using the following methods.

Permethylation

Carbohydrate polymers were permethylated using the method of Laine *et al*. (2002). About 20 mg of ground sodium hydroxide and 0.1 mL of methyl iodide were added to 3–5 mg of the oligo- or polysaccharide sample in 0.5 mL dry dimethylsulfoxide. The sample was kept for 30 minutes in an ultrasonic bath at room temperature. Water was added and the mixture was extracted with dichloromethane (1 mL). The organic phase was separated, re-extracted with water, dried (anhydrous sodium sulfate) and evaporated to dryness with a stream of nitrogen at room temperature.

Methanolysis

Monosaccharides, permethylated carbohydrate polymers and carbohydrate polymers for compositional analysis were hydrolysed and methylated using the methanolysis method of Ciucanu and Caprita (2007). A solution of 2 M HCl/methanol was prepared by slow addition of 16 mL acetylchloride to dry methanol so that the final volume of the mixture was 100 mL. An aliquot of this solution (2 mL) was added to the sample (either from the previous step or 3–5 mg) in a Reacti-vial and sealed with a Teflon seal. The samples were heated in a 'hot-block' at 80°C for 16 h. After cooling, 5 μL of a sorbitol solution (100 mg / 100 mL in methanol) was added to the reaction mixture as an internal standard. The samples were evaporated to dryness using a stream of nitrogen at room temperature.

Silylation

The methylated sugars from the previous steps were silylated prior to analysis using a modification of the SMRI method (Anon, 2005a).

GC-MS Conditions

The derivatised sugars were separated on a Varian CP3800 GC equipped with a CP8400 autosampler. A Varian FactorFour VF-5ms (crosslinked 5% phenyl-methyl siloxane) column (30 m x 0.25 mm ID with DF=0.25 film thickness) was used. Column flow was set to 1.0 mL/min using helium as the carrier gas. The temperature program started with a temperature of 140°C held for 1 minute, a ramp of 2°C per minute to 218°C, followed by a ramp of 10°C per minute to 280°C. The final hold was two minutes. Injection temperature was 280°C with a splitless injection of 1 μL. The transfer line was held at 280°C and the mass spectrometer had a delay of five minutes. Data were collected over a range of m/z 40–650.

Discussion

Basics of a GC-MS

A GC-MS consists of four components (Figure 1). A sample containing a mixture of compounds of interest is separated in a GC column. As the separated volatile compounds elute from the column, they are bombarded with electrons within an ion-source in a process termed ionisation. These energetic electrons cause the compound to disintegrate and produce a mixture of positively charged ions in the gas phase. Provided the ionisation occurs under the same conditions, the disintegration will be reproducible. The resultant mixture of ions is accelerated into an analyser where they are separated and sorted from each other according to their mass-to-charge (m/z) ratio

by manipulation of electrical fields. The sorted ions are collected by a detector and converted to a proportional electrical current. The data system records the magnitude of these electrical signals as a function of m/z and converts this information into a mass spectrum. The three MS components (ion source, analyser and detector) are housed within a vacuum system to minimise interaction with other molecules.

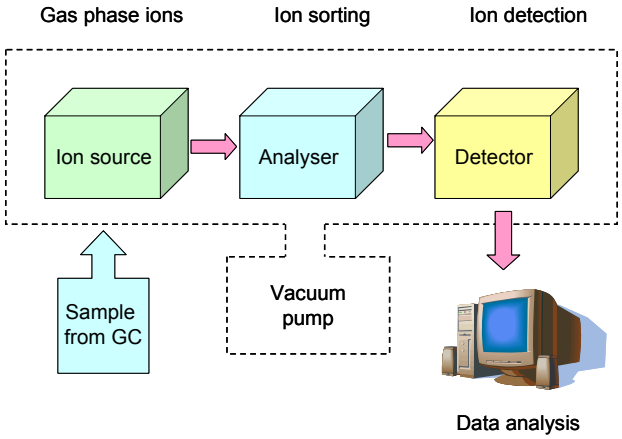

Fig. 1—Diagram of a GC mass spectrometer.

A mass spectrum is a graph of ion intensity as a function of m/z ratio and is often depicted as a simple histogram (shown in Figure 2 for carbon dioxide). This record of ions and their intensities serve to establish the molecular weight and structure of the compound being analysed.

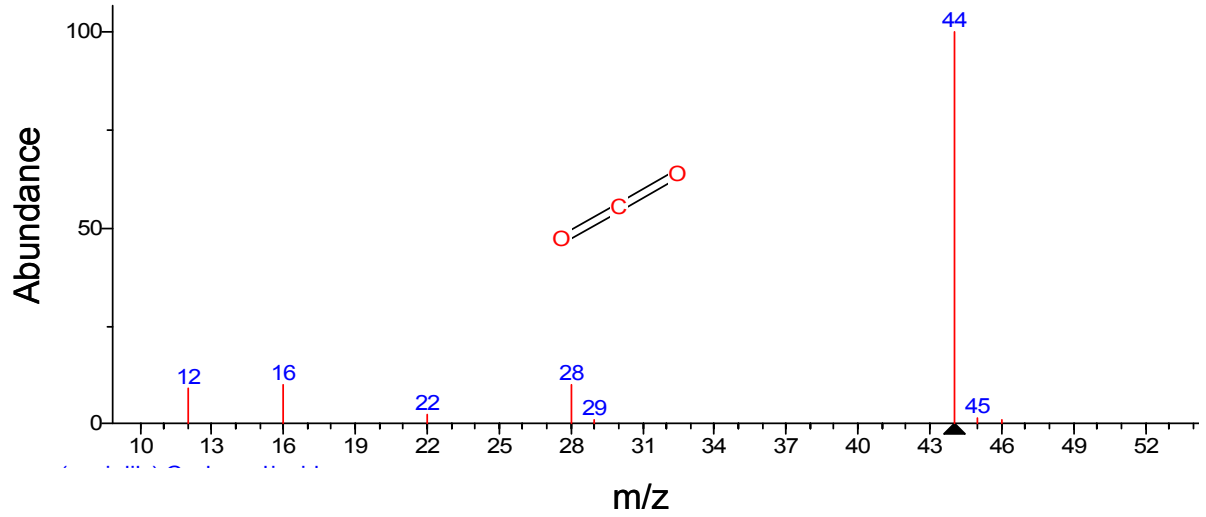

Fig. 2—Mass spectrum of carbon dioxide.

As the CO_2 molecule enters the ion source, the electron beam 'smashes' some of the CO_2 molecules into ionised fragments; all of which are positively charged. The ionised CO_2^+ molecule (known as the molecular ion) appears at m/z 44 (the molecular mass of CO_2). The ionised fragments appear in the spectrum at m/z values less than the molecular ion.

Cleavage of a carbon-oxygen bond in the molecular ion to produce ionised carbon monoxide (CO^+) or ionised atomic oxygen (O^+) results in the fragment ions at m/z 28 and 16 respectively. The loss of two neutral oxygen atoms from the CO_2 results in an additional fragment at m/z 12 for carbon (C^+).

There are different designs of mass spectrometer analysers, including quadrapoles, ion-traps, magnetic sectors and time-of-flight (TOF). They can not only be interfaced with GCs but can be

used as stand-alone instruments, connected to liquid chromatographs (LC-MS) or inductively coupled plasma spectrophotometers (ICP-MS).

Analysis of sugars by GC

A monosaccharide is classified as a triose, tetrose, pentose or hexose according to the number of carbon atoms in the molecule (three, four, five or six). The sugar may have an aldehyde or ketone group attached and these sugars are then termed aldoses or kestoses. Glucose is an example of an aldohexose while fructose is an example of a ketohexose. It can also be shown that monosaccharides can form a ring structure at the aldehyde or ketone. The formation of the ring produces an asymmetric carbon which gives rise to two isomeric forms of the sugar known as the α- and the β- anomers (Figure 3).

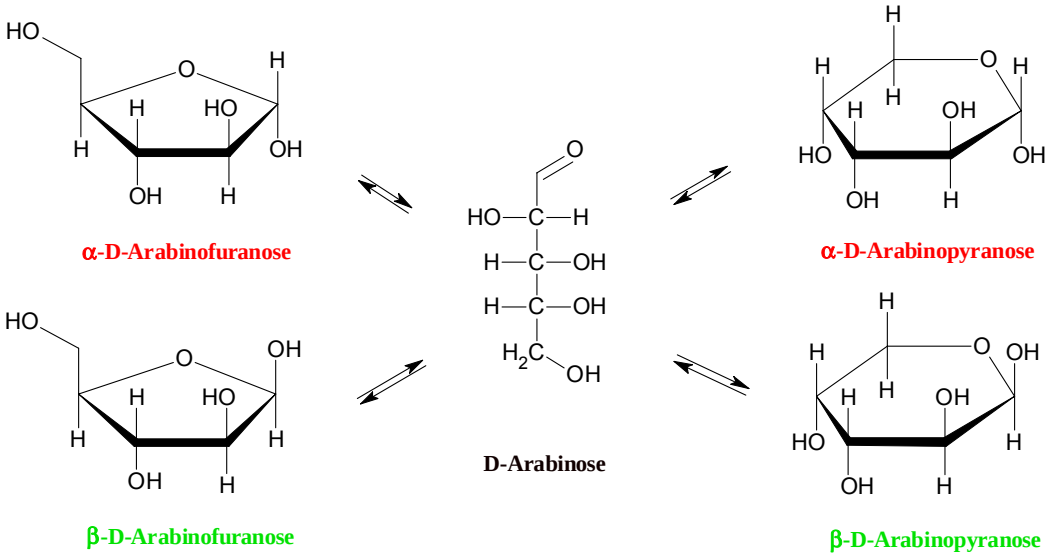

Fig. 3—Anomeric forms of D-arabinose.

A fundamental requirement for the separation and analysis of carbohydrates by GC is that they must be thermally stable and volatile. Carbohydrates must therefore be converted into stable, volatile compounds prior to chromatography. Suitable derivatives can be obtained in a reasonable time using silylation reagents such as hexamethyldisilazane. After silylation, the chromatogram will show multiple peaks of the α- and the β- anomers of the pyranose and furanose rings (Figure 4).

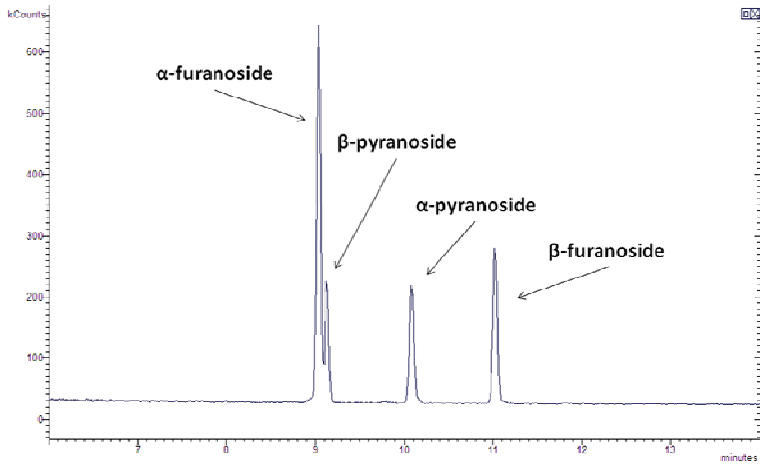

Fig. 4—GC chromatogram showing separation of TMSi arabinose derivatives showing α- and β- anomers of the pyranoside and furanoside forms.

Multiple mass spectra can be taken across each peak as it elutes from the column. The pertinent features of the mass spectra of the TMSi derivatised sugars are seen in Figure 5. No molecular ion at m/z 438 will be seen for the TMSi derivatised arabinose, as carbohydrate molecular ions are generally unstable. Very little difference can be seen between the α- and β-furanoside spectra. These anomers have to be identified based on the difference in retention time on the GC column. However, there is a large difference between the pyranoside compared to the furanoside. The main peak at m/z 217 in the furanoside form is reduced in the pyranoside form, and the peaks at m/z 204 and 191 (not present in the furanoside spectrum) appear in the pyranoside form as seen in Figure 5. This characteristic difference in the mass spectrum can be used to distinguish between furanoside and pyranoside forms of a sugar – the presence of only m/z 217 indicates the presence of a furanoside form of the sugar. The peak at m/z 73 is due to the trimethylsilyl compound that replaced all the active hydrogens in the –OH groups in the sugar during derivatisation. Numerous GC and/or MS studies on TMSi carbohydrates have been reported and the mechanism of fragmentation elucidated (Kochetkov and Chizhov, 1966). Each peak in the mass spectrum can be assigned and used to help determine the structure of the sugar.

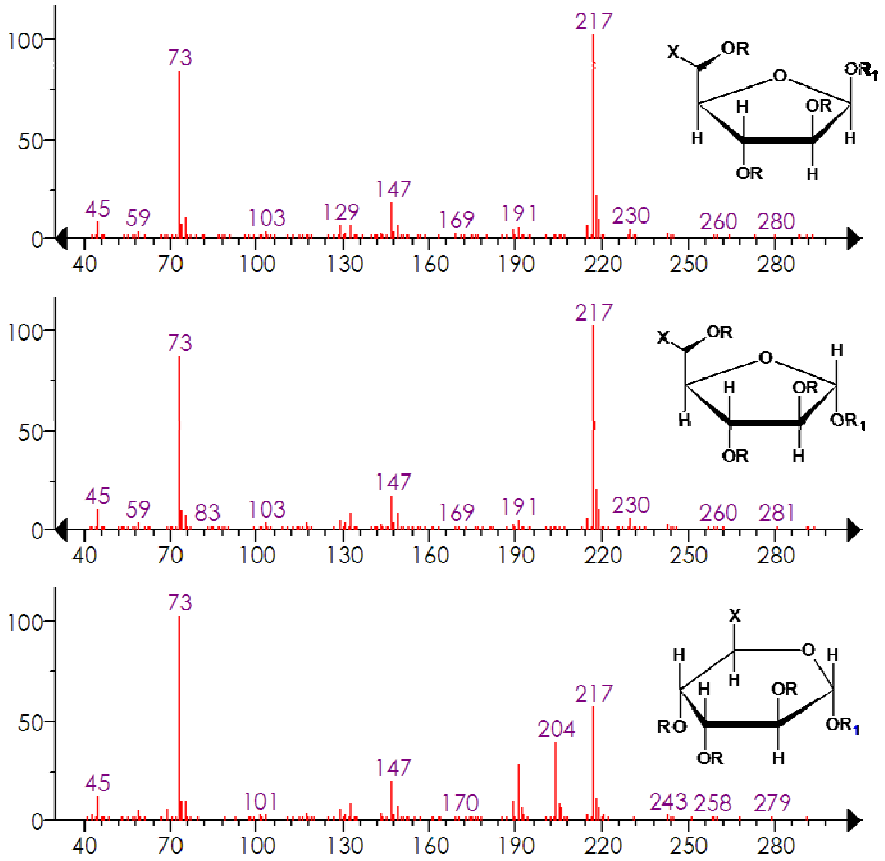

Fig. 5—Comparison of the mass spectra between the β- and α–furanoside forms (upper and middle spectra) and the α- pyranoside form (lower spectra) of TMSi derivatised arabinose. (R = R1 = TMSi; X = H). [y-axis =Abundance, x-axis = m/z]

A direct silylation procedure produces multiple peaks (and thus mass spectra) for each sugar to be analysed. The number of glycoside peaks, their retention times and relative proportions are characteristic of each monosaccharide. If a mixture of sugars is analysed, a rather complex chromatogram results; however, the high efficiency provided by capillary GC columns generally permits satisfactory separations of the peaks to be achieved. Figure 6 shows a typical chromatogram for a mixture of TMSi sugars.

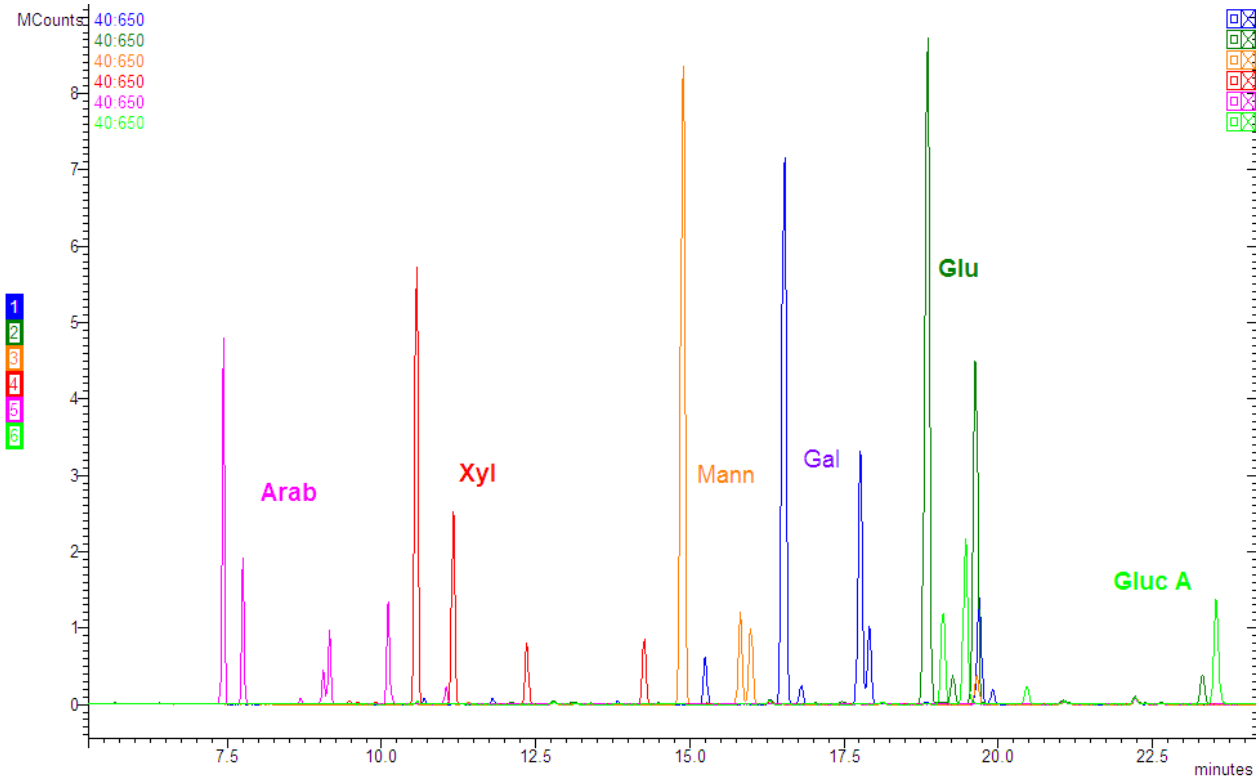

Fig. 6—Chromatogram showing the separation of a mixture of TMSi sugars. Arab = arabinose; Xyl = xylose; Mann = mannose; Gal = galactose; Glu = glucose; Gluc A = gluconic acid.

Compositional analysis of carbohydrate polymers

The analysis of carbohydrate polymers by GC is complicated by the reduced volatility of derivatised polymers.

The very high temperatures required to elute the compounds from the GC column simultaneously degrade and decompose the polymers.

Compositional analysis using GC-MS requires the polymer to be hydrolysed into its constituent sugars which can then be analysed as described.

Both chemical and enzymatic hydrolysis methods exist with the former being the 'brute force' method resulting in a mixture of monosaccharides, while the latter is very specific for a particular linkage and uses milder conditions.

The chemical method is ideal for compositional GC-MS preparation as all the sugars are released. A typical method would use an acidic solution of methanol with the sample being heated at 80°C for 16–20 hours—a method known as methanolysis (Bleton *et al*., 1996).

A methyl group attaches to the carbohydrate hydrolysed from the polymer. After complete hydrolysis, the sample is dried, silylated and analysed.

The resultant chromatogram will show most of the individual carbohydrates that made up the carbohydrate polymer. This process is shown for pullulan in Figure 7.

The mass spectrum of the two peaks shown in Figure 7 confirms that glucose is the only carbohydrate found in pullulan. Note also the presence of m/z fragments at 191, 204 and 217 indicating a pyranoside form of the ring.

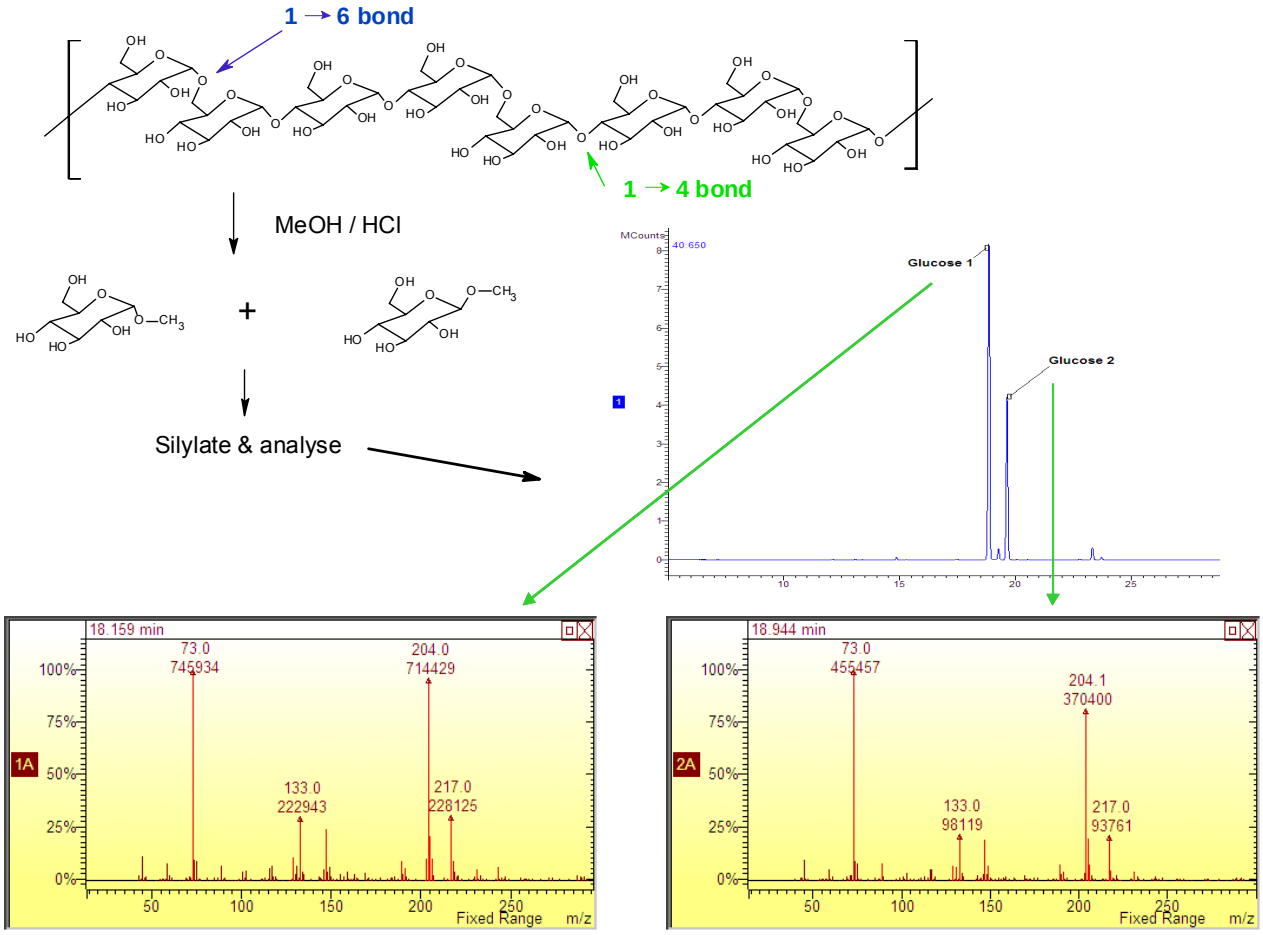

Fig. 7—Methanolysis of pullulan showing hydrolysis and methylation at position 1 and resultant chromatogram after silylation showing only pyranoside glucose peaks.

If the carbohydrate were to contain sugars other than glucose, these would also be seen in the chromatogram and could be identified. An example would be xanthan gum (used to thicken sauces) which has a glucose backbone and a trisaccharide side chain consisting of mannose (with substituents) and glucuronic acid (Figure 8).

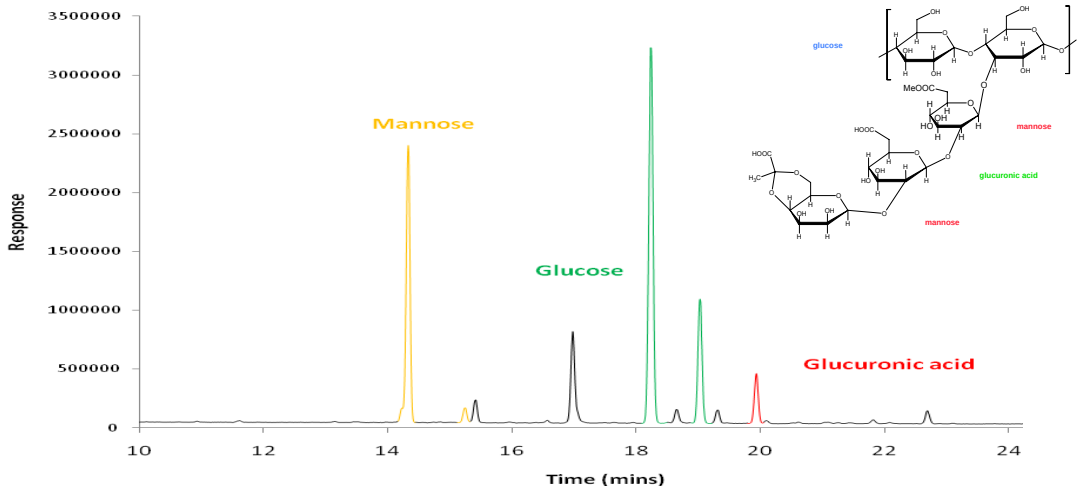

Fig. 8—Chromatogram from the methanolysis of xanthan gum showing the compositional sugars – glucose, mannose and glucuronic acid.

Structural analysis of carbohydrate polymers

The methanolysis method described above will only give the researcher compositional information about a carbohydrate polymer.

When studying the structure of an unknown polysaccharide, several questions arise: how many constituent monosaccharides does it contain, in what order are they linked; does it have a branched or linear structure; are the sugars in the pyranose or furanose form; and are the glycosidic linkages α- or β- or a mixture of the two?

There are many classical methods to determine the linkage between the sugars including methylation, the Barry degradation (Barry, 1943) and the Smith degradation (Smith and van Cleve, 1955) being the most common.

The aim of these techniques is to produce a series of derivatives which requires separation and can be used to identify the ring size and linkage position. These techniques are time consuming and require reasonable quantities of sample for analysis.

The resolving power and sensitivity of GC-MS techniques allows for very small quantities of these derivatives to be prepared and analysed.

The method adopted in our laboratories uses a methanolysis procedure based on the use of methyl iodide in dry dimethyl sulfoxide solution, extraction of the methylated polymer, followed by methanolysis and silylation (Laine *et al*., 2002).

Methylation adds a methyl group to all free hydroxyl groups on the sugar. Methanolysis will hydrolyse the sugar leaving a free hydroxyl group on the sugar which can then be silylated.

The position of the TMSi group will indicate where the sugar was joined to its neighbour. The structure of the sugar containing both methyl and TMSi groups can be determined by GC-MS and the position of the TMSi group will indicate the linkages on the particular sugar.

Dextran is a straight chain glucan consisting of α-1,6 glycosidic linkages between glucose molecules, with branches beginning from α-1,3 linkages (Figure 9, top).

Methylation of the dextran results in all the free hydrogens on the –OH groups being replaced by a methyl group (Step 1).

This is followed by methanolysis (Step 2) to yield the free methylated sugar with the methyl group on carbon 1 being in either the α- or the β- form (shown in the figure as –OMe horizontal on the page).

The other side of the bond now has a free –OH group which can be silylated (Step 3) to form the TMSi derivative.

Note that the glucose that had three other glucose units attached at the beginning of the branch now has two TMSi groups while the terminal glucose on the branch has no TMSi groups.

This results in three derivatised glucose units: 2,3,4-trimethyl-6-TMSi glucose, a lesser quantity of the terminal derivative 2,3,4,6-tetramethyl glucose and very small amounts of the linkage glucose, 2,4-dimethyl-3,6-TMSi derivative.

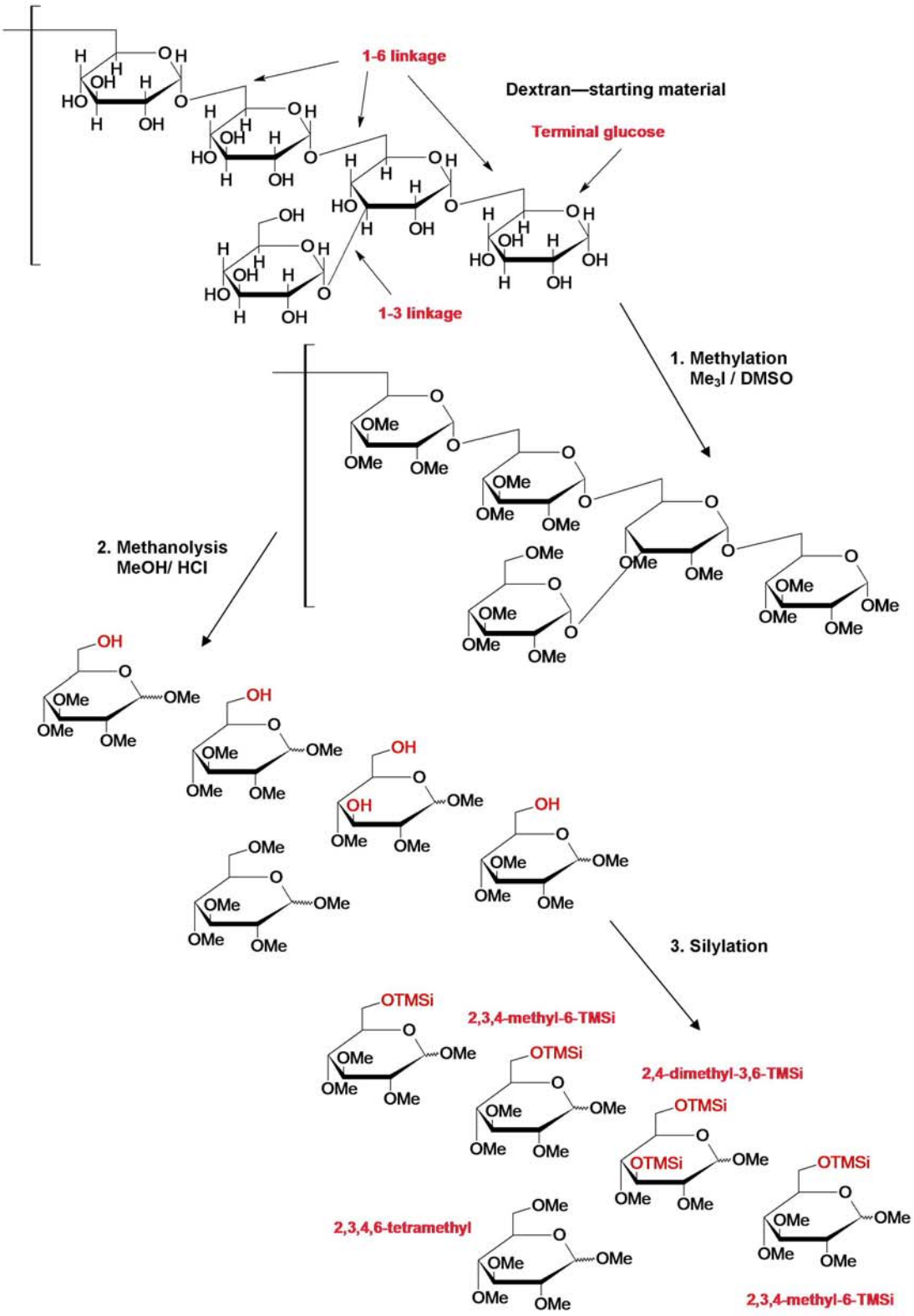

Fig.9—Diagram showing the method of structural analysis using dextran as a model compound. The steps include methylation (Step 1), methanolysis (Step 2) and silylation (Step 3) to produce methylated, TMSi derivatives dependent on the position in the polymer

The resultant chromatogram and two of the mass spectra from this scheme are shown in Figure 10. The mass spectra are quite different and the peaks easily identified.

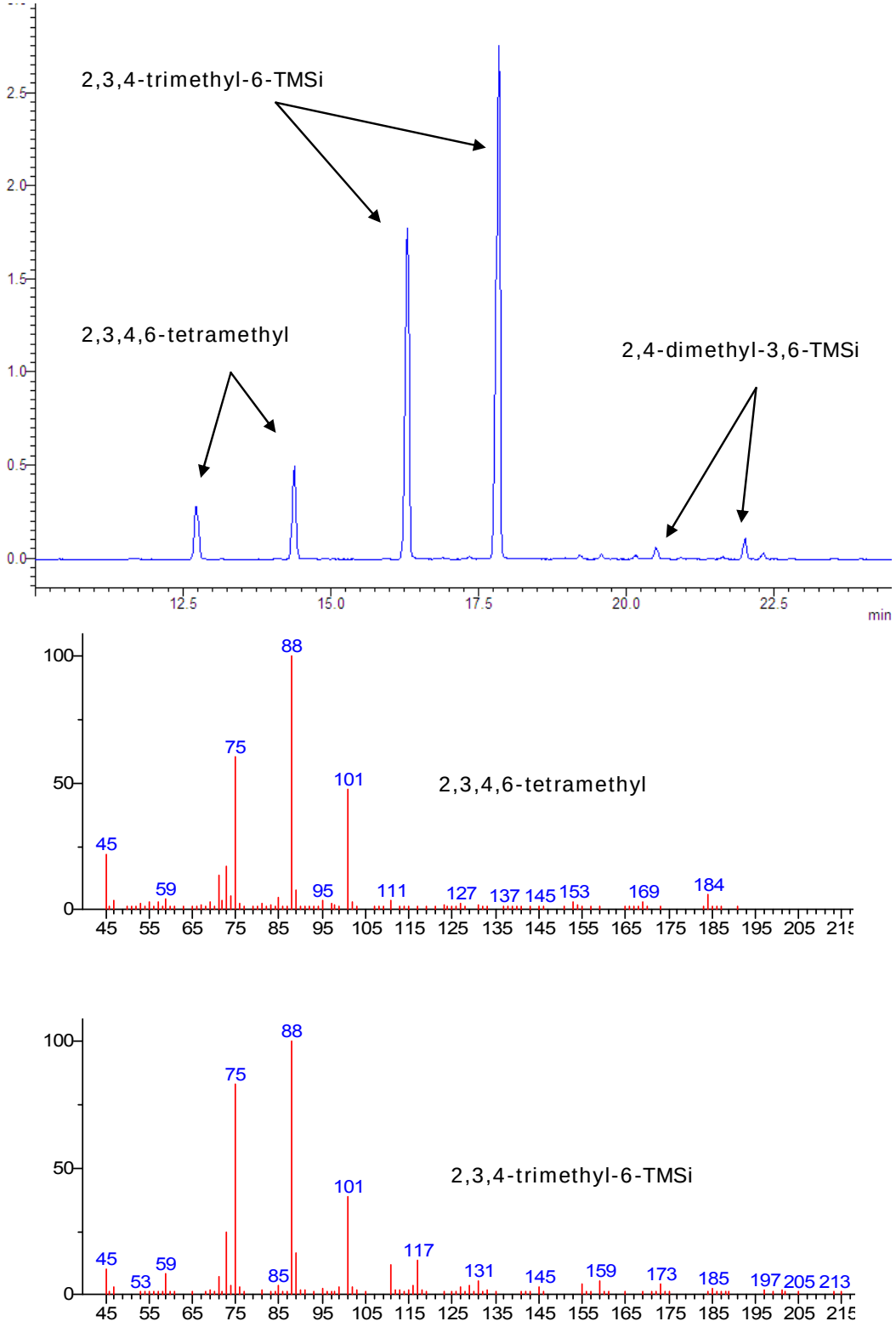

Fig. 10—Chromatogram resulting from the derivatision scheme shown in Figure 9 (top). Mass spectra of the 2,3,4,6-tetramethyl- and 2,3,4-trimethyl-6-TMSi glucose peaks (lower).

Based on the derivatives produced and the height of the peaks, it is possible to postulate that the dextran contains a small quantity of glucose that has linkages on the 3 and 6 position (based on the 2,4-dimethyl-3,6-TMSi derivative), a larger quantity of terminal glucose units (based on the 2,3,4,6-tetramethyl derivative) and most of the polymer consists of 1–6 linkages (based on the 2,3,4-trimethyl-6-TMSi derivative). Quantitation of the peaks is possible giving a ratio of each of the forms present.

In order to determine the retention time and mass spectra of each of the possible linkage combinations, a variety of di- and trisaccharides of known structure can be analysed by the method and a database of results accumulated. Table 1 is an abbreviated list of some of the compounds that can be used as examples for model linkages.

Table 1—Abbreviated list of compounds that can be used to illustrate model linkages for structural composition of carbohydrate polymers (coloured groups indicate different monosaccharides).

Linkage	Methyl position	TMS position	Model compound
Terminal xylose, furanose form	2,3,5	–	Xylose (methylated)
2-linked xylose	3,4	2	Xylotetrose
Terminal arabinose, furanose/pyranose form	2,3,5	–	Arabinose (methylated)
Terminal glucose, furanose form	2,3,5,6		Glucose (methylated)
Terminal glucose, pyranose form	2,4,5,6		Pullulan
3,6-linked glucose	2,4	3,6	Dextran
2,3,4,6-linked glucose		2,3,4,6	Glucose (silylated)
4-linked galactose	2,4,6	4	Lupin
6-linked galactose	2,3,4	6	Stachyose

The practical use of the techniques in carbohydrate research

As part of the strategic research thrust, SMRI has been investigating the phenomenon of refractory (‘hard-to-boil’) massecuites.

Polysaccharides (gums) have been implicated as a possible contributing factor to this problem (Koster *et al,* 1992; Duffaut and Godshall, 2004; Eggleston and Cote, 2008) and, therefore, SMRI researchers wished to study the composition of the polysaccharides in the refractory materials.

The initial study included eight samples of refractory and normal B molasses collected from four South African mills. The massecuites were deemed normal or refractory by mill personnel based on low molasses exhaustions which subsequently resulted in high target purity differences and recovery losses.

Analyses on the samples included haze dextran, gums, starch, consistency, sulfated ash, sucrose, glucose, fructose and brix. The gums were isolated using the SASTA method for gum determination (Anon, 2005b) and the composition analysed using methanolysis and silylation as described.

Selected results are shown in Table 2 while a chromatographic comparison between one of the normal and abnormal massecuite pairs is shown in Figure 11.

Table 2—Results of selected analyses of some refractory B molasses from three South African mills.

Analyte	Mill 1		Mill 2		Mill3	
	Normal	Refractory	Normal	Refractory	Normal	Refractory
Consistency (Pa.s)	0.33	3.31	0.8	3.46	0.58	1.40
Gums (ppm)	13 100	20 100	11 300	19 100	11 300	18 500
Arabinose	3.5	1.7	4.8	1.3	5.1	2.7
Xylose	3.8	2.4	7.1	2.2	7.2	4.2
Mannose	3.9	4.8	4.7	4.7	5.7	7.3
Galactose	15.1	10.1	17.4	9.0	17.3	11.8
Glucose	73.7	80.9	66.0	82.8	64.7	74.0
Gluconic acid	0.0	0.0	0.1	0.0	0.0	0.0

The refractory samples showed both increased consistency and concentration of gums compared to the normal samples. GC-MS analysis of the gums showed that, while all gum samples contained arabinose, mannose, galactose, glucose and traces of gluconic acid, the amount of arabinose, xylose, and galactose decreased in the 'hard-to-boil' samples while glucose and mannose increased. Structural analysis of the gums is continuing in an effort to understand the causes of this phenomenon.

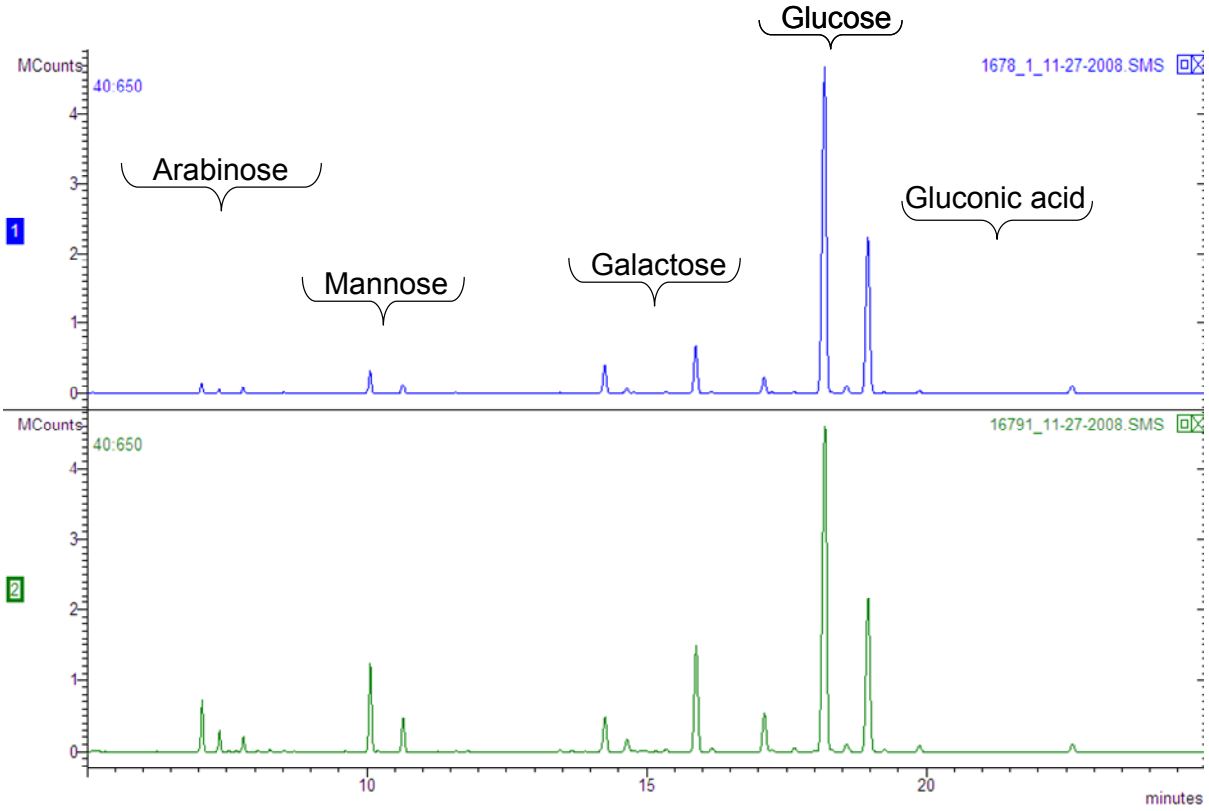

Fig. 11—Chromatograms comparing a normal (upper) and 'hard-to-boil' massecuite (lower).

Conclusion

Gas chromatography, coupled with mass spectrometry as a detection technique, has been shown to be a powerful analysis tool. Although the fundamentals of the technique are easily understood, it is only with modern PC-based equipment that the applications have become commonplace. The application of the chemical derivatisation methods (methylation and

methanolysis), combined with the sensitivity and selectivity of the GC-MS instrument, is allowing a new understanding in research problems of oligomer and polymeric carbohydrate compounds within the SMRI research program and is complementary to other analysis methods.

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L'OUTIL GC-MS POUR L'ANALYSE DES HYDRATES DE CARBONE EN LABORATOIRE DE RECHERCHE

Par

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MOTS CLEFS: Chromatographie Gazeuse, Spectrométrie de Masse, Silylation, Hydrates de Carbone.

Résume

LA CHROMATOGRAPHIE en phase gazeuse associée à la spectrométrie de masse (GC-MS), comme technique de détection, est un outil important pour le chercheur. Les principes fondamentaux de la technique et son application à l'analyse des hydrates de carbone à l'aide de dérivés volatiles tels que les éthers de triméthylsilyl, sont décrits. Les spectres de masse des fragments sont utiles pour élucider la structure des glucides (pyranoside vs furanoside), la nature du saccharide et le type des liens glycosides. L'utilisation de la methylation et de d'hydrolyse (généralement appelé methanolysis), suivie de silylation et de l'analyse par CPG-SM, est une technique utile pour caractériser les oligomère et les matériaux polymères glucides (gommes et films). L'utilisation de ces techniques avec des composés modèles et pour l'un des programmes de recherche du Sugar Milling Research Institute sera discuté.

GC-MS UNA HERRAMENTA PARA EL ANALISIS DE CARBOHIDRATOS A NIVEL DE INVESTIGACION

Por

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PALABRAS CLAVES: Cromatografía de Gases, Espectrometría de Masas, Sililación, Carbohidratos.

Resumen

LA CROMATOGRAFÍA de gases acoplada con espectrometría de masas (GC-MS) utilizada como una técnica de detección, es una poderosa herramienta de análisis para investigaciones químicas. Los fundamentos de esta técnica de análisis así como su aplicación son descritos para el análisis de carbohidratos usando derivados volátiles tales como el trimetil silil éteres. Se presenta el fragmento de un espectro de masas que puede ser usado como un indicador de la estructura del anillo (piranosa vs furanosa) de los carbohidratos, ó para establecer la naturaleza reductora de los sacáridos como también para establecer el tipo de algunos enlaces glicosídicos. El uso de la metilación y la hidrólisis (típicamente conocida como metanólisis) seguido por sililación y análisis GC-MS se presenta como una herramienta útil para caracterizar carbohidratos tales como polímeros y oligómeros (gomas y películas). Se presenta el uso de estas técnicas usando un compuestos típicos y su aplicación en uno de los programas de investigación del Sugar Milling Research Institute.