

# PRACTICAL TRACERS FOR THE SUGAR INDUSTRY — THE ANALYTICAL FEASIBILITY OF USING LITHIUM, CHLORIDE OR POTASSIUM

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## Abstract

Two types of tracer are described — added and natural. The former is useful for following physical and the latter for monitoring chemical pathways in the sugar factory. Some analytical advantages and limitations of using lithium for the first application and chloride or potassium for the second are highlighted.

## Introduction

Tracers in the sugar factory can be used to answer two different questions viz. 'how much goes where for how long?' and 'what happens to it whilst there?' The analytical requirements for the tracer element are different in the two circumstances.

The first case is essentially one of following the physical progress of a portion of process stream in the short-term. This usually involves shock addition to the stream of something normally absent that can be detected precisely at low levels. Accuracy is not necessarily essential, but is useful, if unexpected leakage or recycling is encountered. Sampling frequency is generally high, particularly in the early stages.

The second application calls for a natural constituent present at levels which can be measured readily and reliably. This component should be unaffected by processing conditions and should not be added at significant levels during process. The need here is for both precision and accuracy rather than sensitivity. Compositing over fairly long time intervals is needed to smooth out short-term fluctuations in concentration. Provided these conditions are met other component concentrations can be compared from point-to-point within the factory to evaluate chemical change during processing.

We have been involved in both these aspects of monitoring. Although many points have not been followed to a logical conclusion, we felt it might be useful to record our findings and suggestions and to highlight some of the analytical problems often overlooked in routine application. Although the merits of monitors should be self-evident we have noticed that the concept (particularly in the second context) is not always as well appreciated as it might be.

If this paper generates an awareness of different tracer applications and stimulates interest in improving their usefulness it will have served its purpose.

## Part 1. Lithium

Radio-isotopes (either labelled compounds or encapsulated sources) have been widely used as tracers (Basson<sup>1</sup>, Hirschmuller<sup>2</sup>). There are drawbacks to their application in the food industry on a factory scale (e.g. the need for short half-life isotopes, adequate supervision and economics). In situations where on-line monitoring is essential there may be no alternative. (Smith<sup>3</sup>). Several other compounds have been used for flow pattern tracing and evaluation of equipment design — with varying degrees of success. These include common salt and measurement of sodium (Foster<sup>4</sup>) or

chloride (Matthesius<sup>5</sup>), dyes (Matthesius<sup>5</sup>) and PVC pellets (Strickland<sup>6</sup>).

The Australians (Grimley<sup>7</sup>, Broadfoot<sup>8</sup>, Wright<sup>9</sup>) found lithium a useful alternative. Direct application of the procedure described by Wright and Broadfoot<sup>9</sup> was unsatisfactory. Some of the points to emerge when establishing analytical guidelines for measuring lithium by flame emission are outlined below — generally we tried to identify and eliminate the observed interferences. A paper has since been published by Haysom<sup>10</sup> which is essentially in agreement with our findings.

## Experimental

Standards were prepared using lithium chloride (Riedel-de-Haën for Analysis — min. assay 99%). Initial drying was unnecessary since response did not improve after drying the salt for 3–4 hours at 400°C. Potassium nitrate (BDH — Analar) (1500 mg/l as potassium) was added to all standards.

Samples (to contain less than 2 g solids) were massed, dissolved in de-ionised water, 10 ml of a 15 000 mg/l potassium solution (as KNO<sub>3</sub>) added and diluted to 100 ml. The diluted samples were filtered (S&S 3000) discarding at least 20 ml and the filtrate aspirated directly.

All measurements were made on a Varian AA-175 in the emission mode. The response was optimised for flame height and nebuliser adjustment. Instrumental conditions are shown in Table 1. De-ionised water was aspirated between samples.

TABLE 1

Instrumental parameters for flame emission measurement of lithium in the air-acetylene flame

Mode	Emission
Wavelength (nm) .. .. .	670,8
Slit width (nm) .. .. .	0,2
Integration period (sec) .. .. .	3
Air flow (l min <sup>-1</sup> ) .. .. .	10,2
Acetylene flow (l min <sup>-1</sup> ) .. .. .	1,4

## Results and Discussion

### 1. Analytical range and sensitivity.

For the reliable detection of added tracer the analytical method needs to be capable of high sensitivity coupled with adequate precision.

Aqueous dilutions of lithium chloride were used to determine the sensitivity of the method. Linearity was poor for lithium levels greater than 5 mg/l. However, sensitivity was such that with 100 ug/l full-scale the relative standard deviation (rsd) for 10 samples was 0,5%. At these levels linearity was excellent (linear correlation coefficient  $r = 0,999$ ).

2. Precision.

(a) Integration time.

Integrated signals eliminate short-term noise and so give improved precision. The choice of integration time was limited to 0 or 3 or 10 sec with the instrument used. There was no difference in response or precision with either 3 or 10 sec integration periods (Table 2). Integration periods of 3 sec were used subsequently.

TABLE 2  
Effect of integration period  
(Range 0-350 µg/l)

Integration time	3 sec	10 sec
Response: $y = r$	$-0,8 + 0,280 x$ 0,9995	$-0,9 + 0,280 x$ 0,9996
Precision: Mean rsd (%) for 5 readings at each of 9 Li levels	1,7	1,5
t	1,118	
$t_{95;8}$	2,306	

(b) Stability and calibration frequency.

Flame emission spectrometers are operated in the single-beam mode and thus are sensitive to warm-up drift and medium term fluctuation. After allowing the instrument a 20 minute warm-up period (with the flame alight), a set of lithium standards in the range 0 - 150 µg/l lithium and containing 2 000 mg/l potassium was read at intervals during the following hour (Figure 1). The change in response averaged about one unit (or 1% relative) per 5-minute interval. Hence to reduce baseline drift to less than 2%, at least one re-calibration is essential within each 5 - 10 minute period. With such precautions about 1,5% rsd was achieved routinely.

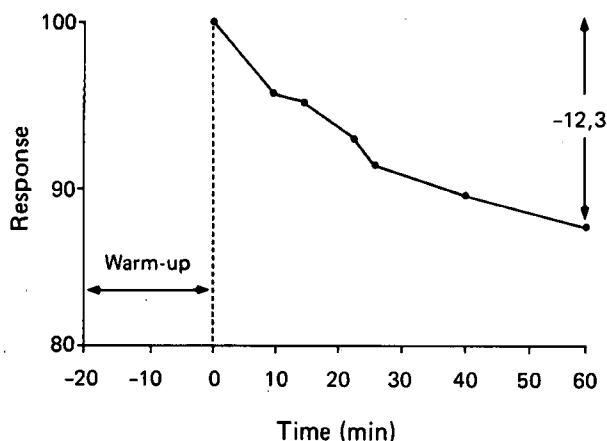


FIGURE 1 Instrumental drift at 670,8 nm with 150 µg lithium/l set full-scale.

3. Sample storage.

A juice sample (with added lithium) was refrigerated and re-analysed in successive runs. Over a six-week period the response dropped progressively from 4,10 mg/kg to 2,60 mg/kg. At the same time freshly prepared juice with added lithium gave the expected response. The stored juice thickened considerably although there was

no significant microbiological activity. Possible explanations are the formation of sucrose-salt complexes or slow formation of LiOH which would not break down appreciably in the air-acetylene flame. We mention this as a cautionary note if samples are to be stored before analysis.

4. Interferences.

Using an analytical range of 0 - 400 µg/l lithium it was evident that low levels of juice increased, and higher levels of juice decreased, the lithium response. Although response was linear for all levels of juice, the scatter was greater at high juice levels. (Figure 2).

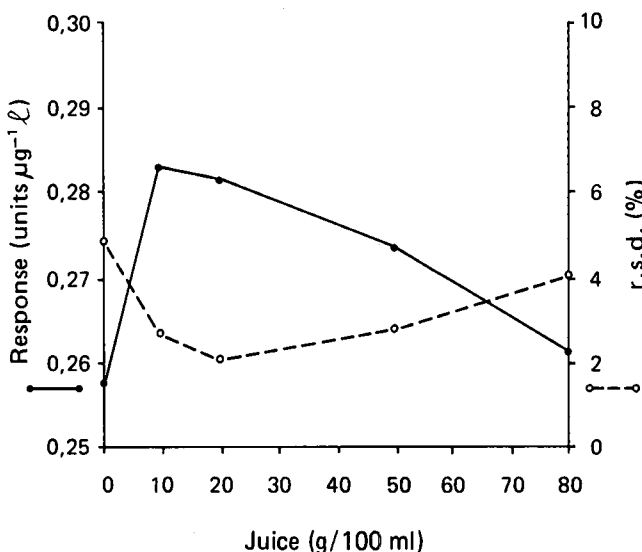


FIGURE 2 Effect of juice concentration on lithium response and precision

In order to maintain the simplicity of direct sample dilution and to avoid extensive matrix-matching we tried to identify and pin-point the interfering effects.

(a) Sucrose.

If juice or standards contained more than 1% sucrose each additional percentage increase in sucrose caused nearly 1% decrease in lithium response. We attributed this to the reduced uptake rate caused by higher viscosities.

(b) Ionisation interference.

Conditions which deplete the atomic concentration cause low response. Many elements ionise easily in hot flames. In cool flames molecular compounds can be formed. We assumed the increased lithium response at low juice levels was probably due to the ready ionisation of elements, such as potassium, preventing lithium ionisation. Excess potassium (1 000 - 2 000 mg/l) can be added to the standards and samples to suppress this lithium ionisation. However, if potassium chloride was used the lithium response dropped by about 4,5% as potassium increased from 500 to 2 000 mg/l. In cool flames chlorides can form HCl, leaving excess hydroxyl ions to combine with lithium as refractory LiOH. The effect could not be overcome by adjusting flame conditions (lean or slightly oxidising). When potassium nitrate was used as ionisation suppressant the change in slope over the range 500 - 2 000 mg potassium/l was only 1,5 - 2,5% at a given sucrose concentration. (Figure 3).

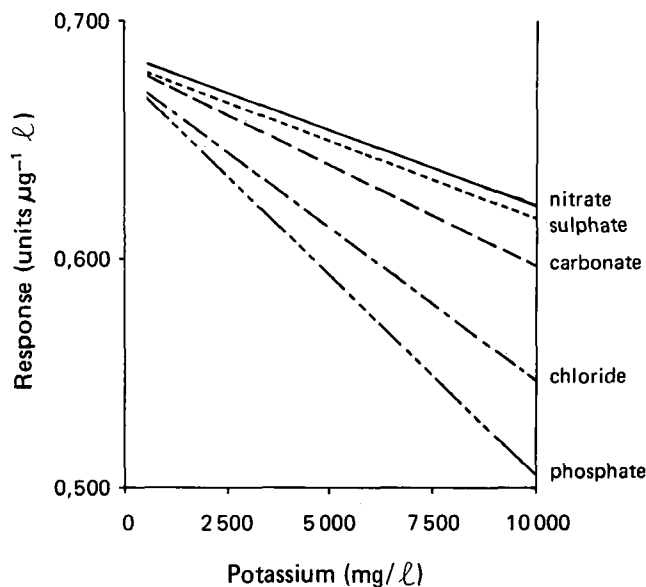


FIGURE 3 Effect of potassium salt on lithium response.

(c) *Chemical interference.*

The extent of interference due to calcium was investigated by adding increasing amounts of calcium (as nitrate) to lithium solutions containing 2% sucrose and either 500 or 2 000 mg/l potassium (as nitrate). The range of calcium evaluated was 0 - 1 000 mg/l. (A solution containing 2,5% molasses would probably have less than 350 mg/l calcium) (MacGillivray<sup>11</sup>) (Table 3).

Two partially compensating effects were noticed —

- (i) as calcium levels increased the background emission increased.
- (ii) as calcium levels increased the calibration slope (or response) decreased.

TABLE 3  
Effect of calcium on lithium response in presence of potassium (500-2 000 mg/l) and sucrose (2%)

Ca (mg/l)	Intercept (c) (units)	Slope (m) (units mg <sup>-1</sup> ℓ)
0	0,2	654
250	1,8	637
500	2,7	638
750	4,5	629
1 000	6,8	622

Linear calibration of the form  $y = c + mx$

A low purity product such as molasses containing 1,5% calcium and no lithium would give a background lithium content of 150 ug/kg and low recovery (97%) of added lithium.

Although analytically unattractive the problem can be avoided by preparing standards using samples collected just prior to a test run. We were not in a position to follow up this problem. Haysom<sup>10</sup> has recently published results indicating that the calcium interference can be overcome by precipitating calcium as the oxalate.

It has since occurred to us that an elegant way of overcoming the chemical interference would be to use the hotter nitrous oxide-acetylene flame, provided ionisation effects can be suppressed (caesium salts might prove more effective than potassium nitrate). The following points are amongst the reasons for this suggestion.

The calcium interference in air-acetylene flames has been attributed to the broad molecular absorption of CaO and CaOH centred on 665,3 nm. Furuta<sup>12</sup> has shown that refractory LiO is a predominant species when aspirating lithium halide into the air-acetylene flame and Bulewicz<sup>13</sup> has shown that in low temperature flames excess chloride reacts with hydrogen to release hydroxyl radicals and to shift equilibrium in favour of molecular LiOH.

Pickett<sup>14</sup> and Hildon<sup>15</sup> have used the nitrous oxide-acetylene flame to measure lithium by emission spectroscopy. They obtained a 7 to 10 fold increase in sensitivity as compared with air-acetylene.

5. *Sample background and recoveries of added lithium.*

With calibration standards containing potassium (as potassium chloride), product streams were diluted to contain 1 - 2% dissolved solids. Lithium chloride was added so that diluted samples contained 0 - 150 ug lithium per l. Results are presented in Table 4.

TABLE 4  
Recovery of added lithium

Product	Conc. (g/100 ml)	Background Li (mg kg <sup>-1</sup> )	Recovery (%)
Juice .. .. .	10,0	0,010	100,2 ± 0,8
Syrup .. .. .	3,0	0,010	96,9 ± 1,0
A-massecuite .. .. .	2,0	0,013	100,5 ± 0,9
B-massecuite .. .. .	2,0	0,042	98,2 ± 1,7
C-massecuite .. .. .	2,0	0,079	97,3 ± 1,2
C-molasses .. .. .	2,5	0,141	93,4 ± 0,5

Part 2. Chloride

Chloride has sometimes been used as an added tracer. However, the relatively high levels in cane lead to high background levels producing imprecise results.

High chloride levels make it a useful natural tracer. The potentiometric method of Comrie<sup>16</sup> is both precise and accurate and has been applied by MacGillivray<sup>17</sup> to detect sugar losses in the factory, and by ourselves (Morel du Boil<sup>18</sup>) to monitor monosaccharide degradation in the boiling house.

Because of the relative simplicity and reliability of the analysis we tried to extend the application of chloride to juice streams. Juice samples were collected routinely and mercuric chloride added to enable accurate pol, sucrose, glucose and fructose analyses to be carried out. The logistics of collecting parallel samples without added preservative were considered impractical.

Juice preservative added to juice samples at the recommended dosage (0,2 ml l<sup>-1</sup>) increases the chloride level in the sample by about 1 - 2%. However, the recommended level is usually exceeded because preservative is added before collecting the sample on the assumption that throughput is constant. It is often believed that if ten drops are adequate then fifty must be better. Consequently the level of added preservative is variable and unpredictable.

Theoretically it is possible to correct for this added preservative and it was felt worth attempting.

Experimental

Titration were carried out using a Metrohm Potentiograph E436 in the differential mode. A Metrohm combined Ag/AgCl electrode EA 246 was used.

**Results and Discussion**

The juice preservative is essentially a mixture of potassium iodide, potassium iodomercurate and potassium chloride. Since silver iodide and silver iodomercurate are considerably more insoluble than silver chloride it is theoretically possible to obtain two end points and thus to distinguish the added chloride, iodide and iodomercurate from the natural chloride. The correction is determined using the same batch of preservative.

In practice correction for added preservative caused a three-fold deterioration in precision. The first end point was over-estimated so that the corrected titre was too low. (Table 5).

**TABLE 5**  
Effect of preservative when added to juice at recommended levels (0,2 ml l<sup>-1</sup>)

	*Titre (ml)	rsd (%)
Juice .. .. .	11,51 ± 0,02	0,18
Juice + preservative .. . . .	11,81 ± 0,02	0,19
Juice (corrected for preservative)	11,31 ± 0,07	0,60

\* Mean for 8 titrations using 0,1 N AgNO<sub>3</sub>  
Bias if uncorrected: + 2,6%  
Bias after correction: - 1,7%

With low titres the effect was worse (Table 6).

**TABLE 6**  
Effect of sample size on titre (20,0 ml burette) for juice with added preservative

Sample (g)	Titre Total (ml) a	Titre first peak (ml) b	b/a (%)	1,93 b/a (%) *	Chloride (mg/kg)
20 .. .	3,17	0,13	4,1	7,9	518
40 .. .	6,37	0,21	3,3	6,4	529
75 .. .	11,81	0,30	2,5	4,9	531
112,5 ..	17,79	0,49	2,3	4,3	531

\* Ratio of chloride to iodide — iodomercurate in juice preservative (stoichiometric preparation) was 0,93.  
These figures represent the correction as a percentage of the total titre.

These effects are partly attributable to co-precipitation of silver chloride with silver iodide (particularly in dilute solutions) and partly to the skewing of the Ag<sub>2</sub> [Hg I<sub>4</sub>] peak due to the divalent iodomercurate anion. Normally these influences are regarded as insignificant.

Another point which bears mentioning is that unless the silver electrode is kept completely free of silver iodomercurate (by immersing in dilute thiosulphate solution between titrations and washing well with water) the response to the first peak drops off rapidly. (Figure 4). If the electrode does not respond to the added preservative, chloride over-estimation results. With a poorly maintained electrode the chloride peak is also affected eventually.

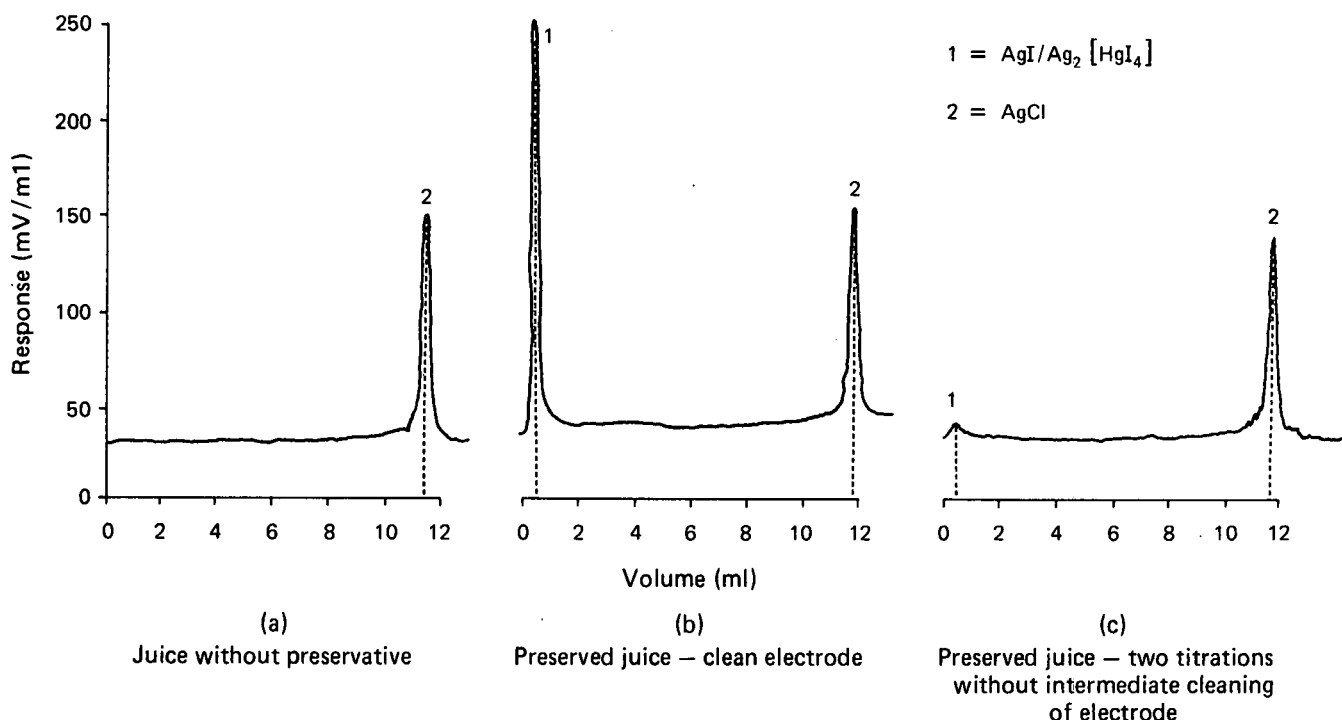
Although chloride is a very good monitor in most areas of the factory it can become unreliable when samples are preserved with mercuric salts.

Results in Table 7 were obtained at Mount Edgecombe during 1978/79. (Juices were preserved with mercuric chloride). As in the previous season (Morel du Boil<sup>19</sup>) there was a significant increase in the F/G ratio throughout the boiling house caused by highly significant glucose drops. However, although both the F/G ratio and fructose showed a significant drop during clarification the unreliability of the chloride measurement in the presence of juice preservative causes reluctance in accepting this result.

The use of mercuric iodide in place of mercuric chloride will avoid the loss of precision encountered when correcting for added chloride. The over-estimation of the first peak can be minimised by using aliquots as large as possible and by standardising under similar conditions i.e. by adding chloride-free preservative or sodium iodide to standard sodium chloride to give similar titres.

**Part 3. Potassium**

In view of the shortcomings of chloride as a monitor for front-end streams (caused by the variable and significant bias introduced by the juice preservative), other possible natural tracers were considered. Potassium and sodium should be as soluble as chloride and as unaffected by processing conditions. After initial work sodium was disregarded (mainly



**FIGURE 4** Effect of juice preservative on electrode response.

**TABLE 7**  
Chloride as monitor for fructose and glucose at Mount Edgecombe 1978/79

Stream	n	F/G	t	*Sig	(F + G) Cl	t	*Sig	G Cl	t	*Sig	F Cl	t	*Sig
MJ .. ..	16	1,12	—	—	9,13	—	—	4,35	—	—	4,79	—	—
CJ .. ..		1,08	2,31	S	8,74	1,67	NS	4,25	0,72	NS	4,50	2,80	S
Syr .. ..		1,06	1,70	NS	8,10	2,12	NS	3,96	1,78	NS	4,15	2,28	S
C-Mol ..		1,53	9,65	HS	7,20	4,11	HS	2,95	10,10	HS	4,27	1,05	NS
Syr .. ..	7	1,06	—	—	6,94	—	—	3,40	—	—	3,57	—	—
A-Mol ..		1,12	5,10	S	6,78	0,86	NS	3,20	1,81	NS	3,57	0,08	NS
B-Mol ..		1,23	5,79	S	6,35	2,33	NS	2,86	3,12	S	3,50	1,04	NS
C-Mol ..		1,65	6,25	HS	5,41	4,81	S	2,09	7,36	HS	3,36	1,56	NS

Results represent n consecutive fortnightly composites

t <sub>95; 15</sub>	2,13	t <sub>95; 6</sub>	2,45
t <sub>99; 15</sub>	2,95	t <sub>99; 6</sub>	3,71
t <sub>99,9; 15</sub>	4,07	t <sub>99,9; 6</sub>	5,96

\* Significance: HS: Significant at the 0,1% level; S: Significant at the 5% level; NS: Not significant.

on account of its extremely low level in some juices — 5 mg/l). Potassium is a major constituent of sugarcane ash and Carpenter and Bichsel<sup>20</sup> have used it to monitor ash constituents in the factory. Atomic absorption techniques for potassium are in general use. Modern instrumentation should give the necessary degree of accuracy and precision. A point to bear in mind is that the conventional juice preservative contains significant amounts of potassium. It was decided to evaluate the reliability of the technique and, if adequate, to recommend suitable and acceptable alterations to the juice preservative.

**Experimental**

*Sample preparation:* Sample dilutions were essentially a compromise to enable both sodium and potassium to be measured with a single sample preparation.

*Standards* were prepared from AR grade KCl. Salts were dried 4 hours at 400°C. CsCl (1 000 mg/l Cs) was added to all standards.

*Juice* was centrifuged at 6 500 g for 15 min (4 g or 8 g) of supernatant were taken, CsCl (10 ml of 10 000 mg/l solution) added and diluted to 100 ml with de-ionised water.

*Syrup* (1,00 g) was massed, CsCl (10 ml of 10 000 mg/l solution) added and diluted to 100 ml with de-ionised water.

*Molasses* (2,00 g) was diluted to 250 ml with de-ionised water. Aliquots (10 ml or 20 ml) were taken, CsCl (10 ml of 10 000 mg/l solution) added and diluted to 100 ml with de-ionised water.

All measurements were made on a Varian AA-475 in the double-beam absorption mode. The burner height and nebuliser were adjusted for optimum response. Instrumental conditions are shown in Table 8. De-ionised water was aspirated between samples.

**Results and Discussion**

**PRECISION.**

(i) *Instrumental:* Statistical concepts such as integration are widely used in modern AA instruments to correct for short-term noise. With the AA-475 short-term precision was better than 0,5% rsd provided integration periods of at least 4 sec were used.

**TABLE 8**

Instrumental parameters for atomic absorption measurement of potassium in the air-acetylene flame

Range .. .. .	0-80 mgℓ <sup>-1</sup>
Mode .. .. .	Double beam
	Concentration, (40; 80 mgℓ <sup>-1</sup> std)
λ .. .. .	769,9 nm
Slit width .. .. .	0,2 nm
Lamp current .. .. .	10 mA
(Na-K dual element)	
Int. hold .. .. .	4 sec
Air (cyl) .. .. .	400 kPa
(rot) .. .. .	7,4
(ℓmin <sup>-1</sup> ) .. .. .	12
Acetylene (cyl) .. .. .	12 psi
(rot) .. .. .	3,4
(ℓmin <sup>-1</sup> ) .. .. .	2

(ii) *Sample:* Relative standard deviations for duplicate samples analysed within the same batch were better than 1%, whereas re-analysis in different batches gave 2% rsd (Table 9).

**TABLE 9**

Precision: Comparison of within-batch and between-batch replicates

	Within-batch		Between-batch Molasses
	Juice	Molasses	
No. pairs .. .. .	37	55	48
Mean diff. .. .. .	15 mgℓ	0,037%	0,103%
Overall rsd (%) ..	0,8	0,7	2,0

**ACCURACY.**

(i) *Instrumental:* The built-in curve-fitting procedures enabled direct concentration readout with about 0,7% relative error over the analytical range 20 - 80 mg/l.

(ii) *Sample:* The potassium content was measured at two different concentrations of juice (4 or 8 g juice/100 ml) and molasses (0,08 or 0,16 g molasses/100 mls). In each case the lower sample concentration gave potassium results 1 - 2% higher. This bias was significant at the 95% level (Table 10).

TABLE 10  
Potassium — bias caused by sample concentration

	Juice	Molasses
Df .. .. .	9	86
$\frac{C_2 - C_1}{C_1}$ .. .. .	21,4 mg/l	0,042%
$\frac{C_2}{C_1}$ .. .. .	1193 mg/l	3,747%
$\frac{C_2}{C_1}$ .. .. .	1214 mg/l	3,788%
$t_{0,5}$ .. .. .	4,657	9,845
$t_{0,5}$ .. .. .	2,262	2,000
$\left(\frac{C_2}{C_1}\right) \times 100$ .. .. .	1,8%	1,1%

Df = degrees of freedom

$\frac{C_2 - C_1}{C_1}$  = mean difference

$\frac{C_2}{C_1}$  = mean value at lower concentration (see text)

$\frac{C_2}{C_1}$  = mean value at higher concentration (see text)

Potassium added to dilute juice or molasses in the range 10 - 45 mg/l gave 98,4% recovery with an rsd of 1,5%. (216 additions).

To enable accurate pol, brix, sucrose, fructose and glucose determinations on juice samples, preservation is essential. Currently mercuric salts are most effective. At present the formulation incorporates both potassium and chloride leading to analytical difficulties in front-end streams if either of these ions is used for in-process balances. The influence of conventional juice preservative added at the recommended dosage will inflate juice potassium levels by about 20 - 25 mg/l — this is about 1,5 to 2,5% bias. As with chloride this addition tends to be variable. With the AA technique there is no simple correction.

For the past few seasons chloride has been measured in syrup and other back-end streams to estimate sugar losses in the boiling house. (Table 7). Recommended preservative dosages will have an insignificant effect on the chloride estimation in syrup. However this preservative addition has tended to be enthusiastic on occasion. Last season the potentiometric mercuriodate peak was used to correct both the potassium and chloride analyses for added preservative. After correction, five monthly comparisons gave a mean potassium to chloride ratio of 1,59 for syrup and 1,60 for molasses, which is encouraging.

It was found that replacing the potassium iodide with sodium iodide had no effect on glc sugar determinations. If the preservative is further modified so that mercuric chloride is replaced with mercuric iodide, both potassium and chloride can be measured directly in juices. Such a preservative formulation is being evaluated at present.

The AA investigation was intended as a preliminary evaluation of the potential and adequacy of potassium as a monitor. In this context it is felt that the precision achieved and the observed deviations from linearity are probably the worst conditions that could be expected. The results are sufficiently encouraging to warrant 'fine-tuning' of the analytical procedure to enable a direct comparison with chloride in the presence of suitable juice preservative.

### Conclusions

Using flame emission lithium can be estimated with good precision in the analytical range 0 - 150 ug/l in the presence of 2% dissolved solids provided standards and samples contain potassium nitrate. With low purity products such as final molasses lithium is underestimated by about 3%. It is postulated that the chemical interference causing this could be avoided if the nitrous oxide-acetylene flame were used. The accuracy and precision obtained make lithium analytically attractive as an added tracer.

Chloride is an ideal natural tracer in most areas of the factory. It can be measured accurately, precisely and simply using potentiometric techniques. However when samples have been preserved with mercuric salts chloride tends to be under-estimated. Despite limitations chloride has been used to indicate sugar degradation within the factory.

Potassium can be determined with about 1% precision using atomic absorption techniques. The preliminary investigation indicated less than 2% bias, which could probably be overcome with larger sample dilutions.

In general potassium (using AA) will be less reliable than chloride. If conventional juice preservative is used potassium is unsuitable since no simple correction is possible and preservative levels are variable. However, if the juice preservative does not include potassium or chloride then potassium and chloride are probably equally effective natural monitors for preserved juices. The comparison should be made using suitably modified juice preservative.

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