*Short Communications/Kort Mededelings*

# Milk iodine determination: The suitability of milk samples collected in the South African Milk Recording Scheme

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*Received 29 September 1986*

Milk samples collected for analysis in the South African Milk Recording Scheme are routinely preserved by the addition of potassium dichromate. The influence of this preservative, the duration of storage temperatures (0°C or room temperature) and effect of temperature prior to readings on the iodine levels of such milk samples was investigated. The only significant finding was that iodine levels declined ($P<0,01$) in milk samples stored at room temperature for extended periods. The results show that milk samples collected for the Recording Scheme may be used for electrometric iodine determinations as a practical screening technique for milk, but care should be taken to avoid contamination of the samples with iodophor teat dips and other sources of iodine contamination.

Kaliumdikromaat word in die Suid-Afrikaanse Melkaantekeningskema gebruik om melk wat vir ontledings gekollekteer is, te preserveer. Die invloed wat hierdie preserveerder, tydperk van opberging, temperatuur gedurende opberging (0°C of kamertemperatuur) en verhitting van melk voor metings op jodiumpeile in die melk het, is ondersoek. Slegs die storing van melk teen kamertemperatuur oor lang tydperke het 'n beduidende ($P<0,01$) verlaging op die jodiumpeil van melk gehad. Die resultate toon dat melkmonsters wat vir die Melkaantekeningskema verkry is, as 'n vinnige keuringsmetode vir jodiumbepalings gebruik kan word. Besoedeling as gevolg van jodiumbevattende speendope en ander bronne van jodium sal egter vermy moet word.

**Keywords:** Milk iodine, dairy cows, milk recording scheme, preservatives, storage

The concentration of iodine in milk can be used to indicate the iodine status of dairy cows (Alderman & Stranks, 1967). Certain areas in South Africa, e.g. parts of Natal and the Cape were found to be iodine deficient (Boyazoglu, 1976). In the South African Milk Recording Scheme, milk from individual cows is collected monthly and tested for butterfat, protein and lactose content (Anonymous, 1984). These milk samples offer a convenient way of obtaining samples for iodine determinations from a wide range of farms, provided that the treatment to which the milk is subjected, does not affect its iodine level. Factors to consider are that the milk is preserved with potassium dichromate ($K_2Cr_2O_7$) and that it is stored for various lengths of time in refrigerators. Prior to analysis for quality assessment, the milk is warmed to 45°C for 10–15 minutes to facilitate better mixing (G. Rew, 1986, Personal communication). An investigation was carried out to establish whether these factors exert any significant influence on the iodine concentration of the milk.

The concentration of iodine in fresh milk was compared with that in duplicate samples which were preserved with $K_2Cr_2O_7$ for different lengths of time under refrigerated conditions or at room temperature. Crystallized $K_2Cr_2O_7$ was measured out in the containers to give a dilution of 0,2% with the volume of milk added. Samples of preserved milk were also held at different temperatures for 30 minutes, cooled and analysed for iodine to determine the effect on iodine concentrations of heat treatments prior to analysis. In further comparisons milk samples were collected from cows (and ewes) at milking and preserved with $K_2Cr_2O_7$. Duplicates of these samples were passed through the normal processes of transportation, storage and infrared analysis at the milk recording laboratory before iodine assay was performed.

The iodide specific ion electrode method of Lacroix & Wong (1980) as described by van Ryssen, van Malsen & van Blerk (1985) was used to determine the iodine concentration in milk. Care was taken to record the readings while the electrodes and the milk or standards were sealed from light (Black, Pasco & Welton, 1980) and the temperature of the milk was *ca.* 25°C.

Analyses of variance and regressions were performed on the results with the use of the statistical package 'Genstat' (Genstat V Mark 4.03C, 1980. Lawes Agricultural Trust, Rothamsted Experimental Station).

Slight differences between readings on the pH meter were amplified when the $Q$ values obtained from a table were multiplied by the concentration of the standards added to the milk. A pH meter with a four digital scale as compared to three as used in this investigation would allow the use of more detailed information from the tables and would have improved the accuracy considerably. However, because of the wide range considered as normal in milk (25 – 300 gI/$\ell$) (Alderman & Stranks, 1967; Harding, 1982) fairly wide differences between readings should not jeopardize the reliability of interpretations.

Potassium dichromate did not have a major effect on the iodine readings in milk (Table 1). After 2 days of storage in the presence of $K_2Cr_2O_7$, an increase ($P<0,05$) of 6,6% was measured above the iodine level in fresh milk. After 0 and 4 days of storage these differences between fresh and preserved milk were not significant. Binnerts (1979) found a low concentration of formalin to be satisfactory for preserving milk for a few days prior to storage in a refrigerator. The addition of formalin to milk resulted in a dramatic drop in the iodine measurements in our laboratory.

Crecelius (1975) reported a similar observation with formalin. Our results show potassium dichromate to be quite an acceptable preservative of milk when the ion electrode method is used for iodine measurement.

**Table 1** Effect of potassium dichromate as a preservative on iodine concentration in milk stored at 0°C

| | Iodine concentration in milk stored for (days) | | |
|---|---|---|---|
| Item | 0 | 2 | 4 |
| No. of pairs | 93 | 68 | 40 |
| Fresh (μg/ℓ) | 215 | 181 | 205 |
| Dichromate (μg/ℓ) | 225 | 193 | 199 |
| Difference from fresh (%) | 4,7 | 6,6 | -2,9 |
| Standard error of difference | 19,33 | 7,91 | 9,67 |
| Significance | NS[a] | 5% | NS[a] |

[a]NS – not significant

**Table 2** Effect of potassium dichromate as a preservative on iodine concentration in milk stored for extended periods of time at 0°C and at room temperature

| | Iodine concentration (μg/ℓ) in milk stored as follows | | |
|---|---|---|---|
| No. of days | 0°C | Room temperature * | |
| 0 | 112,4 | 4547[a] | 1305[a] |
| 7 | 108,4 | 4311[a] | — |
| 14 | 121,1 | — | — |
| 18 | — | 3808[b] | — |
| 21 | 122,3 | — | 1008[b] |
| *SED* | 4,7 | 148,1 | 67,9 |
| No. of samples | 40 | 6 | 6 |

* Within columns values with superscripts a – b are significantly ($P<0,01$) different

** *SED* – Standard error of difference

However, $K_2Cr_2O_7$ will probably interfere when using photometers for iodine determination although this was not tested.

The changes in iodine readings with time for milk which was preserved with $K_2Cr_2O_7$ were not significant (Table 2). However, after storage for 14 days and longer, lump formation in the milk became evident and this resulted in a larger variation in milk samples taken and thus in the iodine measurements. It therefore seems advisable to measure the iodine in the milk as soon as possible after collection. Bruhn & Franke (1978) and Azuolas & Caple (1984) stored milk at -20°C before analysis. However, Craven & Griffith (1977) found a decrease in the iodine level of milk after 2 weeks in a freezer. At room temperature storage of $K_2Cr_2O_7$treated milk resulted in a substantial ($P<0,01$) reduction in its iodine level after 2 weeks, with lump formation in the milk becoming very pronounced (Table 2).

The heat treatment of the milk did not have a significant effect on the iodine level (Table 3). Binnerts

**Table 3** Effect of heat treatment for 30 minutes on iodine concentration of milk containing 0,2% potassium dichromate as preservative

| | | Iodine concentration[a] (μg/ℓ) at Temperature (°C) | | | | | | |
|---|---|---|---|---|---|---|---|---|
| Test | *n* | 25 | 35 | 45 | 60 | 80 | 90 | *SED*[b] |
| 1 | 9 | 91 | 83 | 81 | 78 | 96 | — | 5,1 |
| 2 | 12 | 141 | 138 | 133 | — | 141 | 141 | 2,8 |
| 3 | 28 | 236 | — | 237 | — | — | — | 2,8 |

[a] Trends not significant

[b] *SED* – Standard error of difference

**Table 4** Concentration of iodine in milk collected directly after milking and in duplicate samples recovered after passing through the milk testing laboratory

| | | Iodine concentration[a] (μg/ℓ) | | |
|---|---|---|---|---|
| Source | No. of pairs | Direct | Via laboratory | *SED* |
| Sheep | 10 | 309 | 303 | 11,4 |
| Cow | 9 | 156 | 146 | 6,6 |
| Cow | 10 | 143 | 143 | 8,8 |

[a] Differences not significant

[b] *SED* – Standard error of difference

(1979) found it acceptable to briefly warm milk to 70°C in a water-bath to facilitate proper mixing before iodine determinations. When milk was heated to temperatures of above 70°C, Bruhn & Franke (1978) and Lacroix & Wong (1980) reported dramatic increases in the iodine readings. This was ascribed to the formation of heat-generated sulphydryl groups in milk which interfere with the activity of the iodide electrode. If milk is warmed to 45°C,as done in the milk recording laboratories, no effect on the iodine measurements in milk should be evident. No significant difference in the iodine concentration of milk was observed when milk was collected directly from the farm or at the milk recording laboratory (Table 4).

From the results presented it seems quite feasible to use the milk collected for milk recording in the South African Milk Recording Scheme for electrometric iodine determinations. However, this should be considered primarily as a screening method to establish potential iodine problem situations because Bruhn & Franke (1978) demonstrated that this electrometric method tends to overestimate milk iodine values at low concentrations and underestimates them at high concentrations as compared to the microchemical procedure of analysis. Furthermore, because the volume of milk collected per cow (36 ml) in the milk recording scheme is not sufficient for this method of iodine determination, milk from various cows must be pooled.

This allows for the mixing of milk from different cows which will eliminate individual variations between cows (Alderman & Stranks, 1967). To obtain the best indication of the iodine status of the cows, they should be grouped according to factors such as level of production, feeding regime, etc. Care must be taken that the milk is not contaminated with iodine from iodophors (van Ryssen, *et al.*, 1985), iodine containing cluster rinses or other contaminants. This can be achieved by the use of iodine-free teat dips, sprays and rinsing agents. Thorough washing and drying of udders before milking and the rinsing of milking machine clusters with water is recommended when iodophors are used.

## Acknowledgements

The University of Natal Research Fund and the Department of Agriculture and Water Affairs are acknowledged for financial assistance.

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