

## Electroanalysis of arsenic in guar gum

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Manuscript received 18 August 1997, revised 4 May 1998, accepted 20 August 1998

Micro-levels of arsenic have been determined in guar gum by differential pulse polarography. The detection limit is 10 ppb.

The widespread use of arsenical compounds and concern over arsenic toxicity have led to the development of numerous methods for quantitative analysis of arsenic<sup>1</sup>. Spectrophotometric methods<sup>2</sup> are subject to numerous interferences and do not have required sensitivity. Flame atomic absorption method is relatively less sensitive to arsenic<sup>3</sup>. Arsenic can be determined at trace levels by neutron activation analysis<sup>4</sup>. Electrochemical methods have great sensitivity specially in environmental monitoring<sup>5</sup>. Another advantage is their ability to distinguish oxidation states of arsenic<sup>6</sup>. Arsenite ( $\text{As}^{3+}$ ) is considered more toxic than arsenate ( $\text{As}^{5+}$ )<sup>7</sup>.

Guar gum, a naturally occurring polysaccharide<sup>8</sup>, has large-scale industrial use<sup>9</sup>, which in effect is likely to increase the arsenic contents of industrial waste-water. Therefore, it is desirable to determine arsenic content of guar gum. In the present communication, arsenic has been determined in terms of its oxidation state of  $\text{As}^{3+}$  and  $\text{As}^{5+}$  in guar gum samples employing differential pulse polarography (DPP).

### Results and Discussion

The study of polarographic reduction of arsenic in different media has shown the suitability of sodium oxalate buffer (pH 4.0) for the determination of arsenic in aqueous matrices where a sharp DP peak of  $\text{As}^{3+}$  was obtained at a potential of  $-1.15 \text{ V}$ <sup>10</sup>. The peak current increased with the concentration of arsenic in the range 0.01–14.0 ppm (Fig. 1). The linearity between the concentration and peak current has been observed. Detection limit achieved was 10 ppb.

**Arsenic in guar gum :** DP polarograms were recorded in the potential range  $-1.0$  to  $-1.4 \text{ V}$ . The peak currents were measured at  $-1.15 \text{ V}$  after making blank correction. The samples (100 ml aliquot) was digested with 5% solution of  $\text{Na}_2\text{S}_2\text{O}_3$  (3 ml) for the reduction of  $\text{As}^{5+}$  to  $\text{As}^{3+}$  so as to enable the determination of  $\text{As}^{5+}$  as  $\text{As}^{3+}$  in the form of total arsenic, due to the electroinactivity of  $\text{As}^{5+}$ <sup>6</sup>.  $\text{As}^{5+}$  concentration was determined by subtracting  $\text{As}^{3+}$  concentration from total arsenic.

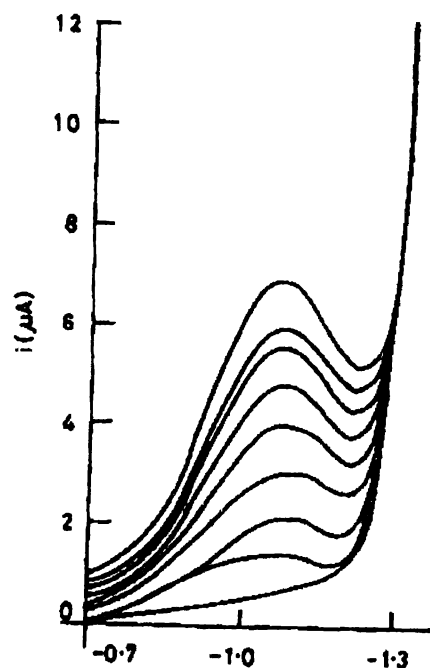


Fig. 1. DP polarograms of  $\text{As}^{\text{III}}$  at varied concentrations 0.1 M sodium oxalate buffer (pH 4.0)

Table 1. Results of arsenic concentration (ppb) in guar gum samples

Sample no	$\text{As}^{\text{III}}$	$\text{As}(\text{total})$	$\text{As}^{\text{V}}$
1	27.22	52.81	25.59
2	20.61	42.81	22.20
3	29.27	54.38	25.11
4	25.94	52.64	26.70
5	20.75	38.95	18.20
6	17.58	32.84	15.26
7	27.40	52.45	25.05
8	30.02	57.21	27.19
9	12.50	42.10	29.60
10	17.11	39.22	22.11
11	23.60	40.71	17.11
12	20.19	47.02	26.83
Average	22.68	46.09	23.41
Standard deviation (S.D. $\pm$ )	5.49	7.6	4.5

Quantitation in all observations was made by standard addition method<sup>11</sup>. Results of analysis of guar gum samples are given in Table 1. The total As (in ppb) values obtained by DPP method was compared with that obtained by atomic absorption spectroscopic method (in parenthesis) : 51.81 (51.99); 42.91 (41.29); 32.84 (30.81).

### Experimental

Guar gum sample was dissolved in 1% sulphuric acid by boiling at 70° for 4 h. The solution was filtered and treated with an oxidising mixture of nitric acid and sulphuric acid to remove biological materials, then evaporated to dryness and dissolved in 0.1 M sodium oxalate for polarographic determination of arsenic.

The chemicals used were of A.R. grade.

A polarographic analyzer (174-A) with a drop timer (174/70) and x-y recorder (RE0074) (EG & G, U.S.A.) was used to record DP polarograms. The settings for DPP were as follows : modulation amplitude, 50 mV; clock time of pulse, 1 s; pulse duration, 57 ms; scan rate, 5 mV s<sup>-1</sup>. Potential was measured against a saturated calomel electrode. A dropping mercury electrode and a platinum wire were used as working and auxiliary electrodes respectively.

An atomic absorption flame spectrometer (Shimadzu AA-6300) was used. All experiments were carried at 25±1°. The solutions were deaerated by passing nitrogen, which was purified by bubbling through a vanadous chloride scrubbing solution for 20 min, prior to the polarographic measurements.

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