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## Novel miniaturized sensors for potentiometric batch and flow-injection analysis (FIA) of perchlorate in fireworks and propellants

Saeed H. M. A. Almeer,<sup>a</sup> Ibrahim A. Zogby,<sup>a</sup> Saad S. M. Hassan<sup>b\*</sup>

<sup>a</sup>Central Laboratory Unit, Qatar University, P.O. Box 2713, Doha, Qatar

<sup>b</sup>Department of Chemistry, Faculty of Science, Ain Shams University, Cairo, Egypt

### Abstract

Three planar miniaturized perchlorate membrane sensors (3 x 5 mm) are prepared using a flexible Kapton substrate coated with nitron-perchlorate (NT-ClO<sub>4</sub>) [sensor I], methylene blue-perchlorate (MB-ClO<sub>4</sub>) [sensor II] and indium-porphyrin (In-Por) [sensor III] as electroactive materials in PVC membranes plasticized with 2-NPPE. Sensors I, II and III display near-Nernstian response for  $1.0 \times 10^{-5}$  -  $1.0 \times 10^{-2}$ ,  $3.1 \times 10^{-5}$  -  $1.0 \times 10^{-2}$  and  $3.1 \times 10^{-6}$  -  $1.0 \times 10^{-2}$  mol L<sup>-1</sup> ClO<sub>4</sub><sup>-</sup> with lower detection limits of  $6.1 \times 10^{-6}$ ,  $6.9 \times 10^{-6}$  and  $1.2 \times 10^{-6}$  mol L<sup>-1</sup>, and anionic calibration slopes of  $50.9 \pm 0.4$ ,  $48.4 \pm 0.4$  and  $57.7 \pm 0.3$  mV decade<sup>-1</sup>, respectively. Methods for determining perchlorate using these sensors offer many attractive advantages including simplicity, flexibility, cost effectiveness, wide linear dynamic response range (0.1 – 1000 ppm), low detection limit ( $< 1.2 \times 10^{-6}$  mol L<sup>-1</sup>  $\equiv$  0.1 ppm), small sample test volume (100  $\mu$ L), safety, short response time ( $< 20$  s), long life span ( $\sim 8$  weeks), and extended wide working pH range (4.5 – 8.0). The sensors show high selectivity in the presence of some inorganic ions (e.g., PO<sub>4</sub><sup>3-</sup>, SO<sub>4</sub><sup>2-</sup>, S<sub>2</sub>O<sub>3</sub><sup>2-</sup>, NO<sub>2</sub><sup>-</sup>, NO<sub>3</sub><sup>-</sup>, N<sub>3</sub><sup>-</sup>, CN<sup>-</sup>, Cl<sup>-</sup>, Br<sup>-</sup>, I<sup>-</sup>) and automation feasibility. Indium-porphyrin based membrane sensor (sensor III) is used as a detector in a wall-jet flow injection set-up to enable accurate flow injection analysis (FIA) of perchlorate in some fireworks without interferences from the associated reducing agents (sulfur and charcoal), binders (dextrin, lactose), coloring agents (calcium, strontium, copper, iron, sodium), color brighten (linseed oil) and regulators (aluminum flakes) which are commonly used in the formulations. The sensor is also used for perchlorate assessment in some propellant powders. The results fairly agree with data obtained by ion-chromatography.

**Keywords:** Miniaturized planer sensors; Perchlorate; Potentiometry; Indium-porphyrin; Nitron; Methylene blue; Fireworks; Propellants, PVC membrane; Flow-injection analysis.

\*Corresponding author. Tel.+201222162766; fax +0224831836

E-mail address: saadsmhassan@yahoo.com (S.S.M.Hassan)

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