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Determination of melamine in milk powder using UPLC coupled to APCI-MS/MS. A new method using ultra-performance liquid chromatography (UPLC) coupled to atmospheric pressure chemical ionization (APCI) tandem mass spectrometry (MS/MS) was developed for the determination of melamine in milk powder. The method involves a simple sample preparation, including the extraction of the analytes from milk powder using acetonitrile, followed by filtration and then the derivatization with 1-fluoro-2,4-dinitrophenylhydrazine. The chromatographic separation was performed on an ACQUITY UPLC BEH C8 column and the analysis of melamine in milk powder was

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performed in negative ionization mode with the following multi-reaction monitoring (MRM) transitions:  $m/z$  108  $\rightarrow$   $m/z$  81 for melamine and  $m/z$  108  $\rightarrow$   $m/z$  93 for its D3-d10 internal standard. The method was validated in terms of linearity ( $r^2 \geq 0.99$ ), sensitivity (LLOD/LLOQ: 0.05/1 mg kg<sup>(-1)</sup>), selectivity (RMSEC of 0.23 mg kg<sup>(-1)</sup>), accuracy (90-120%), precision (0.49-0.60% R.S.D.) and recoveries (99.7-102.6%). The method was applied to the analysis of melamine in milk powder samples from the national market, which showed that melamine at concentrations between 0.5 and 1 mg kg<sup>(-1)</sup> was present in these samples.

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