Analysis of 'poppers' products and analytical detectability of a single use of 'poppers'

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Aims: Alkyl nitrites, also known as 'poppers', have a long history as medical drugs and 'aphrodisiacs'. Typical effects include vasodilation, hypotension, methaemoglobinaemia and reduction of smooth muscle tonus. To our knowledge analytical verification of 'poppers' use by detection of the corresponding alcohols was only described in post mortem cases so far. We aimed to develop a method for detection of a modest 'poppers' dose and apply it to samples taken after inhalation of "Rush Ultra Strong" containing isopropyl, isobutyl, n-pentyl and 2methylbutyl nitrite. In addition, several 'poppers' products were analysed to determine their ingredients. Methods: Analysis was performed on a Clarus gas chromatograph using a capillary column (RTX[®]-502.2, Restek, 60 m, 0.53 mm ID, 3 µm film thickness) fitted with a headspace autosampler and a flame ionisation detector. Carrier gas was H₂ at a flow rate of 45 mL/min. A split flow of 10 mL/min was applied for analysis of body fluids, to analyse the pure nitrites and 'poppers' products the split flow was increased. Results: 76 different 'poppers' flasks were analysed. In 51.3 % of the products the declared contents differed from the analytical results. After modest use of 'poppers' 2-Methyl-1-butanol could be detected in the first 3 serum samples. 1-Pentanol was only found in the second sample (11 minutes after the inhalation), whereas isopropanol and isobutanol could not be detected. Concentrations were below 0.05 mg/L. Conclusions: Recreational use of 'poppers' is detectable by analysis of the corresponding alcohols only for relatively short time after consumption. Co-administration of alcoholic beverages could lead to overlap with typical congener alcohols like isobutanol and 2-methyl-1-butanol. In forensic case work 'poppers' flasks found at the scene should be analysed because they may contain other alkyl nitrites than the ones declared.

1. Introduction

Amyl nitrite was the first alkyl nitrite used for treatment of angina pectoris in the late 19th century due to its effect of vasodilation. Over the years it was misused as 'aphrodisiac', and a variety of alkyl nitrites was found in 'poppers' products [1]. Intoxication cases or fatalities due to methaemoglobinaemia [2,3] or circulatory failure are rare.

2. Material and Methods

Material and Sample Preparation: 500 μ L serum was added to a 20 mL headspace vial containing 1 g sodium sulfate and 600 μ L water. For product screening 76 different 'poppers' flasks were purchased via various online shops. A 500 μ L aliquot of the liquid was transferred to a 20 mL headspace vial. After sealing the vial, 10 μ L of the gas phase was transferred in a second 20 mL headspace vial for analysis.

Methods: Analysis was performed on a Clarus gas chromatograph using a capillary column (RTX[®]-502.2, Proprietary Crossbond[®] diphenyl/dimethyl polysiloxane phase, Restek, 60 m, 0.53 mm ID, 3 µm film thickness) fitted with a headspace autosampler and a flame ionisation detector. Carrier gas was H₂ at a flow rate of 45 mL/min. A split flow of 10 mL/min was

applied for body fluids. For analysing pure nitrites and 'poppers' products the split flow was adjusted to 500 mL/min.

Single use self-experiment: A 44-year-old male volunteer sniffed "Rush Ultra Strong" containing isopropyl, isobutyl, n-pentyl and 2-methylbutyl nitrite. Blood samples were taken prior, during, and after the sniffing (Table 1).

3. Results and Discussion

Product screening: 76 different 'poppers' flasks were analysed. The declared contents differed from the analytical results in 51.3% (39 flasks) of the products. In addition to the declared alkyl nitrites (isopropyl nitrite, isobutyl nitrite or amyl nitrite) butyl nitrite, isopentyl nitrite and 2-methylbutyl nitrite were detected. In 54% (41 flasks) more than one alkyl nitrite was present. Most mixtures contained either a butyl nitrite (butyl nitrite and isobutyl nitrite) or a pentyl nitrite, isopentyl nitrite or 2-methylbutyl nitrite) mixture, presumably due to low purity of the involved chemicals.

Single use: After modest use of 'poppers' ("Rush Ultra Strong") 2-methyl-1-butanol could be detected in the first 3 serum samples. 1-Pentanol was only found in the second sample (11 minutes post inhalation), whereas isopropanol and isobutanol could not be detected. Concentrations were below 0.05 mg/L (Table 1).

Tab. 1.	Experimental	Set up	(time in	ı minutes,	concentrations	in	mg/L);	LODs:	2-methyl-1-butanol	and	1-
pentano	ol 0.01 mg/L, is	sobutano	l and iso	propanol 0).05 mg/L.						

		2-Methyl-1-butanol	1-Pentanol	Isobutanol	Isopropanol			
<i>t</i> = -10	blank	< LOD	< LOD	< LOD	< LOD			
t = 0	4 Sniffs							
<i>t</i> = 2	1 Deep inhalation with holding the breath							
<i>t</i> = 5	Blood sampling	0.02	< LOD	< LOD	< LOD			
<i>t</i> = 9	4 Sniffs							
<i>t</i> = 11	Blood sampling	0.05	0.03	< LOD	< LOD			
<i>t</i> = 21	Blood sampling	0.01	< LOD	< LOD	< LOD			
<i>t</i> = <i>31 / 41</i>	Blood sampling	< LOD	< LOD	< LOD	< LOD			

4. Conclusions

Recreational use of 'poppers' is detectable by analysis of the corresponding alcohols only for relatively short time after consumption. Co-administration of alcoholic beverages could lead to overlap with typical congener alcohols like isobutanol and 2-methyl-1-butanol. In forensic case work 'poppers' flasks found at the scene should be analysed because they may contain other alkyl nitrites than the ones declared.

5. References

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