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Chemical Analysis of Oleoresin Capsicum Products

Institute of Environmental Chemistry
National Research Council Canada

TECHNICAL REPORT
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SUMMARY

Aerosol cans from three manufacturers of the Oleoresin Capsicum(OC) products (Capstun, Safstun and Peppermace) were examined by various chemical techniques in order to determine the contents.

The results of this examination are compared with the manufacturer supplied MATERIAL SAFETY DATA SHEETS (MSDS). The quantitative results are found in the summary results table. The ozone depletion factor was considered and the density of OC was obtained for each sample.

The contents of the three Oleoresin Capsicum products were in agreement with the MSDS sheets.

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PROCEDURE - For each sample, the following procedure was followed:

1. Attaching sampling tubing to the spray nozzle of the cans. In some cases, this required modification or partial dismantling of the nozzle assembly in order to attach round sampling tubing. Silicone sealant or epoxy glue was used to make a leak tight connection.

2. All samples were weighed after any nozzle modifications and prior to releasing any contents.

3. The cans were then clamped in an inverted position and the sampling tubing was connected to an evacuated 1 L glass sampling cylinder. For one sample, (CAPSTUN), a gear clamp was placed around the trigger assembly and then slowly tightened until a puff of propellant gas was expelled into the glass cylinder. For the other samples, a system of shims and a small lab jack was used to open the can valve to obtain a gas sample.

4. The glass sampling cylinder was then attached to a vacuum rack to facilitate the transfer of a sample of the propellant gas to an infrared gas sampling cell. This was used to obtain the infrared spectrum of the propellant gas.

5. A water bubbler system was then attached to the can sampling system in place of the glass cylinder and the trigger/valve was cautiously opened until a steady flow of gas was observed in the bubbler.The valve was held open until the flow of bubbles ceased thus depressurizing the can.For the CAPSTUN sample, the water also trapped the alcohol vapour and permitted a Gas Chromatographic analysis to confirm the identity of the alcohol.

6. After each sample had depressurized, the can was weighed to determine the weight of propellant lost and a small nail hole was punched in the can bottom to release any residual pressure. A large tubing cutter was used to cut the bottom of the can off (while the can was still clamped in an inverted position).

7. The contents of the can were then poured into a tared beaker and the carrier solvent was evaporated. The beaker was re-weighed and the amount of OC determined.The initial sampling anddepressurization was carried out inside a glove bag inside a fume hood and plastic gloves were worn at all times to prevent skin contact with the OC.

RESULTS

The qualitative findings will be presented for each sample followed by a summary table.

CAPSTUN

PropellantGas: about a 2:1 molar ratio of propane:isobutane determined by comparison to individual standards of propane and isobutane (Figs.1 a,b;2a,b;3a,b)

Carrier : isopropanol.
determined by infrared vapour spectrum and confirmed by gas chromatography

Note - No other alcohols were seen by either technique.

SAFSTUN

Propellant Gas : a mixture of chlorodifluoromethane (HCFC 22) and 1-chloro-1 ,1-difluoroethane (HCFC 142b) (Figs.4a,b) determined by comparison of IR vapour spectrum to literature spectra. No standards were available to determine the molar ratio.

Carrier : the two HCFC's present also act as carriers as they are liquids under pressure in the can

PEPPER MACE MK IV (small can) MK IX (large can)

Propellant Gas : probably compressed air or possibly compressed Nitrogen. The IR vapour spectrum showed only Freon 113 which is a liquid with a low vapour pressure and a boiling point of 47C other gaseous propellants are CO2 and N20 but these have strong distinctive IR spectra which were not observed (Figs.5a,b)

Carrier : Freon 113 (1 ,1,2-trichloro-2,2,1-trifluoroethane)

QUANTITATIVE RESULTS

		CAPSTUN	SAFSTUN	PEPPER MACE SMALL	PEPPER MACE LARGE
can + contents	g	66.0	261.8	27.0	577.1
can	g	28.0	50.6	12.3	85.5
Net weight	g	38.0	211.2	14.7	491.6
OC weight	g	1.14	16.4	0.75	24.6
OC weight	g	3.0	7.8	5.1	5.0
prop. weight	g	16.4	192.5	ND	13.6
prop. weight	g	43.2	91.1	ND	2.8
carrier weight	g	20.46	none	ND	453.4
carrier weight	g	53.8	none	ND	92.2

Note: Carrier weight determined by difference
ND means not determined

OZONE DEPLETION -

CAPSTUN

The hydrocarbon propellants and the alcohol present are not ozonedepleting.

PEPPER MACE

The Freon 113 is severely ozone depleting (Ozone depletion potential (ODP) is 0.8 the ODP of Freon 11 and Freon 12) and it is a controlled substance under the terms of the Montreal Protocol.

SAFSTUN

The two Freons used in this material have ODP's which are about 1 /10 the ODP of Freon 11 and Freon 12 but are not controlled substances at this time.

MATERIAL SAFETY DATA SHEETS(MSDS) -

In general, the contents are in agreement with the MSDS Sheets.Some differences are discussed below.

CAPSTUN

The MSDS sheet for this product only identifies isobutane and "alcohol". The actual propellant gas for this sample was a mixture of isobutane and propane.The "alcohol" was isopropanol which agrees with the printed label information. No methanol, ethanol or higher alcohols were seen in the sample. Our observed OC content of 3 % (by weight) does not agree with the label value of 5.5 % but it is not clear what units are used i.e. weight/volume % or vol/vol %. Our results are slightly low due to a small unavoidable loss of the OC during sampling.

SAFSTUN

There are no major differences with the MSDS sheet except for a difference in the OC content. Our results show 7.8 % (by weight) ,theirs 5 % but again the units are not specified. Our result again is slightly low due to sampling losses.

PEPPER MACE

A notable difference observed here is that no mention is made of the propellant gas used in this product. A high pressure warning is on the label of the MK IX Magnum Fogger but it is not mentioned in the MSDS sheet. Perhaps as the material is compressed Nitrogen 'or more likely compressed air it does not have to be identified. A calculation using the ideal gas law and the volume of this can showed that the pressure could be as high as 300 psig based upon the weight loss of 13.6 g observed after depressurizing the can. Our results showed the OC content to be about 5% (by. weight) for both samples whereas the sheets state 1.5%. Again the units are unclear. In addition, the large can contained one extra ingredient - a dead insect!

For all these samples, the apparent disagreement in OC contents can possibly be attributed to type of units used to express this value. It is possible that the MSDS values and/or label values are using % by volume units i.e. mL OC in mL carrier. A direct comparison cannot be made as these volumes cannot be measured by the sampling method used. Some estimates could be made by using the densities of the carrier solvent and the measured weights but this was not done. In addition, the density of the OC resin is not known and hence the volume of it cannot be determined by calculation. It could be determined by transferral to tared graduated cylinders after the removal of the can bottoms but this was not done.

OTHER OBSERVATIONS

An infrared spectrum is included for each of the OC material isolated from the samples (Figs. 7,8,9). It can be seen that all three are virtually identical. Without having known OC or a reference IR spectrum for comparison one can only assume that this is indeed Oleoresin Capsicum. The OC could be dissolved in methanol, ethanol, isopropanol, and hexane but the best solvent for cleaning it from equipment etc. was a mixture of 50:50 chloroform and ethylene dichloride.

The IR spectra obtained from varius sample scans and some additional information about Ozone depletion are included for your examination.

PART 2 - Examination of Stun Sprays - Density - March 27, 1992

In an attempt to further understand the MSDS sheets information presented with these samples, some additional tests have been performed on these samples. This was to determine the density of the OC collected from the cans.

Values were obtained for the Pepper Mace and Safstun samples but not the Capstun sample (due to a lack of sample). The density value allowed calculation of the volume of OC collected and hence several types of concentration units could be calculated to see if any agreed with the MSDS value. For some calculations the gas law was used to estimate propellant volumes and or pressures. Not all the details of the calculations will be presented here, just a summary of the results.

PEPPER MACE

Property		Large Value	Small Value	Comment
Label net weight	g:	520.0	0.6 OZ	equals 17 g. (for small can)
Observed net weight	g:	491.6	14.7	measured
Observed OC weight	g:	24.6	0.75	measured
Propellant weight	g:	13.6	ND	measured
Carrier weight Fl13	g:	453.4	13.95	calculated (by difference)
Carrier volume	g:	288.8	8.9	calculated
Can volume	g:	545	26	measured
OC volume found	g:	28.3	0.86	calculated
OC density	g/mL:	0.87	0.87	measured
ocwt%g	%:	5.0	5.1	calculated
OC wt/vol Fl13	%:	8.5	8.4	calculated
OC vol/vol Fl13	%:	9.8	9.7	calculated
OC vol/vol can	%:	5.2	3.3	calculated
MSDS OC	%:	1.5	1.5	units not described
MSDS % Fl13	%:	98.5	98.5	units not described
Fl13 g/g label wgt	%:	87.2	82.0	calculated
Fl13 g/g obs'd wgt	%:	92.2	94.9	calculated

Note : The weight of the Freon 113 in the small Pepper Mace is slightly high as it includes the propellant gas weight which was not separately determined.

SAFSTUN

Property		Value	Comment
Label net weight	g:	150.0	
Observed net weight	g :	211.2	measured
Observed OC weight	g :	16.4	measured
Propellantweight	g:	192.6	measured
Can volume	mL:	251.0	measured
OC volume found	mL:	16.1	calculated
OC density	g/mL:	1.012	measured
OC wt% g OC/g nt wgt	% :	7.8	calculated
OC vol/vol can	%:	6.4	calculated
OC wt/vol can	%:	6.5	calculated
MSDS OC content	%:	5.0	units not described
MSDS % volatiles	%:	--	not mentioned
Observed % volatiles	% :	91.1	calculated

Note: No calculations can be made about the volumes of the two Halogenated Hydrocarbon propellants/carriers as the molar ratio of the two gases could not be determined due to a lack of pure samples of these gases.

CAPSTUN

Property			Value
Comment			
Label net weight	1 oz	45.6 mL	1 OZ avdp equals 26.3 g
Observed net weight	g:	38	measured
Observed OC weight	g:	1.14	measured
Propellantweight	g:	16.4	measured
Carrier weight	g:	20.46	calculated (by difference)
Carrier volume	mL:	26	calculated
Can volume	mL:	62	measured
OC volume found	mL:	1.14	estimated
OC density	g/mL:	1.0	estimated by comparison
OC wt% g OC/g nt wgt	%:	3.0	calculated
OC wt/vol ISOPOH	%:	5.6	calculated
OC vol/vol ISOPOH	%:	3.7	calculated
OC vol/vol can	%:	1.2	calculated
MSDS OC content	%:	5.5	stated on can, not sheets
MSDS % volatiles	%:	>95	
Observed % volatiles	%:	97	calculated