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Identifying odours in pighouse air

Up until now it is not possible to conduct continuous and objective precise evaluation of polluting odour intensity from agricultural sources. On the one hand the odour-active compounds involved are to a great extend still unknown and, on the other, the measurement technology is not yet sufficiently sensitive. In this report a new concept is presented which uses a combination of instrument-analytical and olfactometric methods to identify the odour pollution causing compounds in pighouse air.

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Literature

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Rising population density and increased expectations on air quality mean the objective determination of unwished-for odours and their intensity has become an important task in environment-technological research.

In LANDTECHNIK, and in many other cases, results of odour measurements from livestock housing have been publicised with mostly so-called olfactometry used. Continuous recording with chemo-sensors has also applied which, through correlation with olfactometric data, lead to so-called „odour monitoring“ [1]. Whilst both methods are very suitable for the recording of odours they produce parameter totals with which it is not possible to characterise the compounds responsible for the livestock housing smell.

At the German Research Institute for Food Chemistry in Garching a concept has been developed over the past 20 years that allows compounds also recognisable by humans in odour sources (food, grain, thermally-treated raw materials, cooking gases) to be determined [2]. In the following report this method uses the example of pighouse air in the identification of individual strong odour compounds. Such results could deliver important information for future new odour measurement systems.

Materials and method

Pighouse
The investigated building on the research farm contained two conventionally managed 200 pig compartments which were fully stocked during the trial. Feeding was wet mash and slurry was removed via flushing system.

Chemicals
The compounds 1, 2, 5 and 8 given in *table 1* were used as reference compounds by the company Aldrich (Steinheim).

Capillary gas chromatography/olfactometry (GC/O)
GC/O was conducted with a 5160 gas chromatograph (Carlo Erbe, Hofheim) with capillary columns DB-5 and DB-FFAP (30 m • 0.32 mm, 0.25 µm film thickness, J & W Scientific, Folsom, USA) used. The samples were given at 40 °C on the columns. After 2 minutes the temperature was raised 6 °C/min. to 230 °C and maintained for 5 min. The flow rate of the helium carrier gas was 2 ml/min. As depicted in *figure 1* the gas stream was divided at the end of the capillary column (1:1 v/v) and channelled into a flame ionisation detector (FID) and sniffer opening [3].

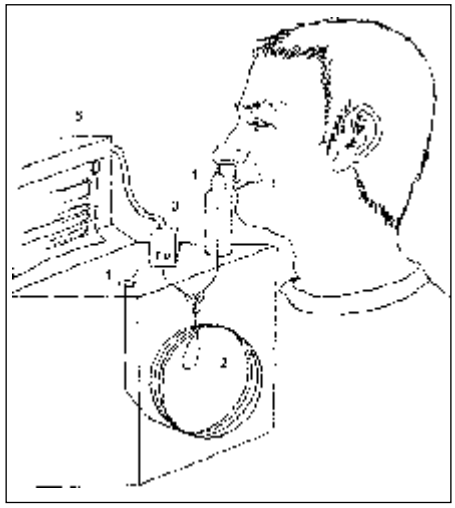

Fig. 1: Scheme of a high resolution gas chromatography/olfactometry equipment: 1 injector, 2 capillary column, 3 FID, 4 sniffer opening, 5 recorder

Capillary gas chromatography/massspectometry (GC/MS)
The massspectrometry investigation was carried out according to [4].

Aroma extract dilution analysis (AEVA)
The pighouse air extract (see sample) was concentrated in a Vigreux column (40 • 1 cm) with micro-distillation [4] to 0.1 ml. The concentrate was gradually diluted with dichlormethane (1:2, v/v) and all dilutions investigated by GC/O with the above-described capillary columns [2].

Results

Sampling
For investigating the odour-active compounds through GC/O an extract containing all volatile compounds in a sample was required. The equipment shown in *figure 2* was developed and fitted in a pighouse for this

task. The equipment comprised a wash bottle (1) filled with solvent (2; as absorbent) and linked with a second glass bottle (3). After closing the valve (4) and cooling the trapping flask with liquid nitrogen (5) condensation causes the development of a slight vacuum of air in (3) whereby pighouse air is drawn into and mixed with the solvent via the inlet tube of the wash bottle. The mixture then enters the trapping flask (3) where the solvent and the volatile compounds from the pighouse air are frozen out. Through this, a continuous volume stream of around 0.3 l/min is reached, confirmed by flow recorder (Q-Cal)). A total of 50 l of pighouse air was condensed for the investigation.

By opening valve 4 and removing the liquid nitrogen the frozen-out air is removed. The frozen extract is then thawed and mixed with the remaining solvent from wash bottle 2. This extract is then concentrated and then used for the analysis of the odour-active components in the following way.

Aroma extract dilution analysis

For GC/O differentiating of odour-active and odourless compounds, the volatile components of the extract are separated in a capillary column *(fig. 1)*. The compounds appearing at the capillary end are simultaneously channelled through a flame ionisation detector (FID) with recorder which records the GC-chromatogram, and a sniffing opening where the eluting solvent flow is sniffed by a person (GC/O). Through this combination of capillary gas chromatography and olfactometry (GC/O) nine regions could be localised during the CG process in which odours appeared *(table 1)*. The odours were described as faecal (1 – 4), sweaty (5 a/b, 8), garlic-type (6), vinegar-type (7) and woody (9).

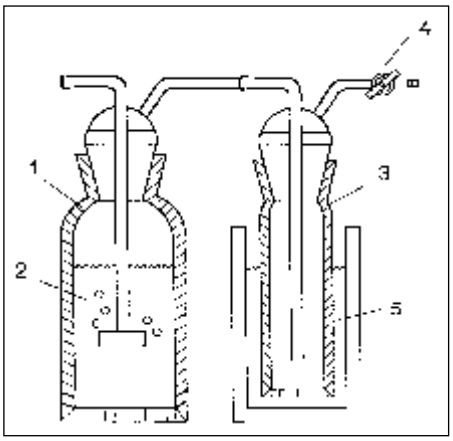

Fig. 2: Apparatus to isolate volatiles from pigsty air. Washing flask (1); solvent (2); trapping flask (3); vent (4) and liquid nitrogen (5)

Because it was not possible to judge the relative odour intensity from smelling the original extract [2], the extract was gradually diluted and the solutions opened to further GC/O analyses until none of the odours were able to be smelled. In this way the odour intensity of a compound was given as FD- (flavour dilution) factor and defined as the highest dilution level where the odour can still be recognised [2].

The results *(table 1)* show that the compounds 1 and 2 could still be recognised in a very high dilution (FD 256) allowing the assumption that these components play an important role in the pighouse odours. Identification experiments indicated that these odour components are 4-methylphenole (p-cresol) and 4-methylindol (scatole). With FD factors of 64 and 16, two further faecal-smelling compounds were (3, 4) detected with structures which have not been able to be explained up until now. The odours 5 – 8, for which the FD factors 4 and 8 were determined, could be identified as dimethyltrisulphide, 2- and 3-methyl butyric acid and acetic and butyric acids. The structure of compound 9 remains open.

Table 1:Results of the aroma extract-diluting analysis of a pighouse air extract

Nr.	Compound[a]	Odour quality	RI[b]		FD[c]
			FFAP	DB-5	
1	4-Methylphenol (p-cresol)	faecal	2071	1078	256
2	3-Methylindol (scatole)	faecal	2475	1395	256
3	Unknown	faecal	2206	1163	64
4	Unknown	faecal	2094	1107	16
5a/b	2-/3-methylbutyric acid	sweaty	1652	-	8
6	Dimethyltrisulphide [d]	like garlic	1348	-	4
7	Acetic acid	like vinegar	1443	-	4
8	Butyric acid	sweaty	1612	-	4
9	Unknown	woody	2541	1447	4

a The compounds were identified through comparison of the retention indices on capillary columns DB-5 and DB-FFAP, the mass spectres (EI) and the odour quality with the properties of the reference substance

b Retention index (RII) determined on the stationary phases FFAP (free fatty acid phase) and DB-5 (silicon duraband-5) according to [5].

c Dilution (FD: flavour dilution-) factor.

d The MS signal of the compound was too weak for a definite interpretation. The compounds were, therefore, identified on the basis of the remaining criteria (vide foot notes).

Conclusions

Through aroma extract dilution analysis, through which instrumental analysis and olfactometric methods are combined, nine odours could be detected in the air sample from a pighouse and their relative contributions to the total odour estimated. The pighouse smell was dominated by faecal notes through the compounds 4-methylphenole and 3-methylindol *(figure 3)*. Detected as further odour active compounds were short-chain carbon acids and dimethyltrisulphide.

The results show that the applied methods allow agricultural-source odours to be reduced to a limited number of compounds which are mainly responsible for an odour or odour pollution. These odours are then suitable as indicators, allowing in future the detection and identification of unwished-for emissions.

Literature

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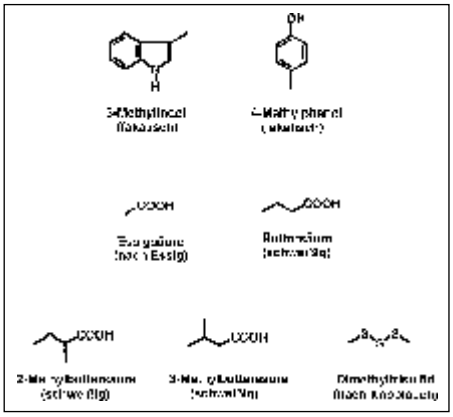

Fig. 3: Odorants identified in pigsty air