Committee for Risk Assessment (RAC)

Opinion

on an Annex XV dossier proposing restrictions on

Inorganic ammonium salts

ECHA/RAC/RES-O-0000005359-66-02/D

Adopted

3 March 2015

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Opinion of the Committee for Risk Assessment

on an Annex XV dossier proposing restrictions of the manufacture, placing on the market or use of a substance within the EU

Having regard to Regulation (EC) No 1907/2006 of the European Parliament and of the Council 18 December 2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (the REACH Regulation), and in particular the definition of a restriction in Article 3(31) and Title VIII thereof, the Committee for Risk Assessment (RAC) has adopted an opinion in accordance with Article 70 of the REACH Regulation on the proposal for restriction of

Chemical names:	**Inorganic ammonium salts**
EC No.:	Not relevant
CAS No.:	Not relevant

This document presents the opinions adopted by RAC. The Background Document (BD), as a supportive document to both RAC and SEAC opinions, gives the detailed ground for the opinion.

PROCESS FOR ADOPTION OF THE OPINION

France has submitted a proposal for a restriction together with the justification and background information documented in an Annex XV dossier. The Annex XV report conforming to the requirements of Annex XV of the REACH Regulation was made publicly available at: ***http://echa.europa.eu/web/guest/restrictions-under-consideration*** on ***18 June 2014***. Interested parties were invited to submit comments and contributions by ***18 December 2014***.

ADOPTION OF THE OPINION OF RAC

Rapporteur, appointed by RAC: ***Agnes SCHULTE***

Co-rapporteur, appointed by RAC: ***Yvonne MULLOOLY***

The RAC opinion as to whether the suggested restrictions are appropriate in reducing the risk to human health and/or the environment has been reached in accordance with Article

70 of the REACH Regulation on ***3 March 2015*****.**

The opinion takes into account the comments of interested parties provided in accordance with Article 69(6) of the REACH Regulation.

The RAC opinion was adopted ***by consensus***.

OPINION

RAC has formulated its opinion on the proposed restriction based on information related to the identified risk and to the identified options to reduce the risk as documented in the Annex XV report and submitted by interested parties as well as other available information as recorded in the Background Document. RAC considers that the proposed restriction on ***inorganic ammonium salts*** is the most appropriate EU wide measure to address the identified risks in terms of the effectiveness in reducing the risks provided that the conditions are modified.

The conditions of the restriction proposed by RAC are:

Substance	**Conditions of restriction**
Entry [#]. Inorganic inorganic ammonium salts	1. ***Articles*** *containing cellulose mixtures treated with inorganic ammonium salts, intended for the purpose of insulation shall not be placed on the market or used, after dd/mm/yyyy[1]" where the release of ammonia from the article in a 24 hour period, during the duration of the test[2] would result in an emission of ammonia greater than 3 ppmV (2.12 mg/m³).* 2. *Cellulose* ***mixtures*** *treated with inorganic ammonium salts intended for the purpose of in situ insulation, shall not be placed on the market or used after "dd/mm/yyyy"[2] where the release of ammonia in a 24 hour period during the duration of the test would result in an ammonia concentration greater than 3 ppmV (2.12 mg/m³).* *The technical specification documentation and any associated packaging, as relevant, should clearly indicate the conditions of use including the maximum loading rate permitted of the cellulose mixture, given in density and thickness, to comply with the maximum 3 ppmV (2.12 mg/m³) emission limit for ammonia in a 24 hr period.* 3. *By way of derogation to point 2 above, mixtures of cellulose insulation treated with inorganic ammonium salts which are only used for the manufacture of cellulose insulation articles do not have to comply with the 3 ppmV (2.12 mg/m³) emission limit of ammonia where it can be shown that the article placed on the market or used has been*

[1] Transition period to be fixed following discussions at SEAC.

[2] Test/test method to be confirmed by CEN. The Commission confirmed their intension to develop, by the entry into force of this regulation, technical specifications for the testing of mixtures or articles containing cellulose treated with inorganic ammonia salts under standard room parameters (size, ventilation) at 90% relative humidity for a period of at least 14 days were followed.

	tested and complies with paragraph 1.

JUSTIFICATION FOR THE OPINION OF RAC

INTRODUCTION

The proposal is to limit the concentration of ammonia that is emitted in any 24 hour period from insulation mixtures or articles containing cellulose treated with inorganic ammonium salts to 3 ppmV (2.12 mg/m^3), rather than to limit the type or quantity of inorganic ammonium salt that can be used to treat cellulose.

The Forum's preference is to set a concentration limit for the content of inorganic ammonium salt found in the insulation material. However, based on the information submitted in the dossier and through the public consultation, RAC is not in a position to establish such a concentration limit due to lack of information on the contributions of solubility, pH, and temperature to any subsequent emissions.

Therefore, the justification for RAC's approach is that information is not currently available on the type, stability or quantity of the various inorganic ammonia salts nor the water content of the insulation material that would be required to substantiate a restriction based on a content limit for ammonium salts. RAC considers that a group entry for inorganic ammonium salts specifying an emission limit for mixtures of cellulose insulation and for insulation articles containing cellulose treated with inorganic ammonium salts is appropriate.

Form the available information, RAC considers that the relative humidity is the main key environmental factor that contributes to the release of ammonia from treated cellulose insulation and the testing of such materials or articles needs to be conducted under standard room conditions (which should be defined in a technical specification of the standard test method) with the exception of relative humidity which should be set at 90%.

In addition, as the final conditions of use of such materials and articles will vary from Member State to Member State (for e.g insulation 'R' or 'U' ratings vary across MSs) it is particularly important for cellulose mixtures that the conditions of use are laid down in technical specifications and packaging labels as relevant.

For any articles manufactured from cellulose mixtures treated with inorganic salts intended as insulation articles, it is the final article that is required to comply with the restriction. The obligation will be on those placing on the market cellulose mixtures treated with inorganic ammonium salts or insulation articles made from cellulose treated with inorganic ammonium salts, to develop a stable mixture or article that achieves this requirement.

IDENTIFIED HAZARD AND RISK

Targeting of the information on hazard and exposure

Cellulose insulation is primarily used to insulate attic spaces (90%) and internal walls of buildings rather than floors or the external walls of such premises.

There are two primary categories of cellulose insulation used and placed on the market:
(1) loose fill material (mixture) that is blown into the area or space to be insulated and
(2) compressed cellulose which is sold as rolls or in rigid, semi rigid panels of insulation (articles).

It is estimated that up to 12% of cellulose insulation is composed of a blend of flame retardants and antifungal compounds while the remaining 85-90% of the material is composed of cellulose fibres from recycled materials such as paper, transport boxes, phone books etc.

RAC is not aware of the use of cellulose mixtures treated with inorganic ammonium salts in composite integration/construction panel type insulation articles, soley intended for outdoor exterior use (e.g. cladding). RAC considers that cellulose insulation articles containing inorganic ammonium salts, soley intended for outdoor exterior use, are unlikely to result in exposure to the indoor environment, although specific evidence for this is lacking.

RAC also considers that cellulose insulation treated with inorganic ammonium salts intended for use on the interior surface of an exterior wall or within the cavity area between the internal and external wall can result in exposures to the indoor environment and is therefore not considered the same as an article intended for outdoor exterior use.

Insulation is an important market outlet for recycled cellulose materials. Currently, across the EU, boric compounds are the primary substances used to treat cellulose insulation material to achieve the specifications for flame retardant requirements.

Following the classification of boric acid as toxic to reproduction Category 1B under CLP, in 2011, the French Authority (CCFAT/DHUP Direction of habitat, urban planning and landscapes) took the decision to no longer issue technical approvals, for the use of boric salts in cellulose insulation materials in France. As a result, the cellulose insulation sector suddenly changed to inorganic ammonium salts (in powder form) as an alternative flame retardant with limited experience in the treatment process. By the end of 2012, 20,000 homes in France were insulated with cellulose insulation that had been treated with inorganic ammonium salts.

Information on hazard(s)

Complaints and reports of smells in homes resulted in the French Authorities undertaking investigations which detected ammonia in homes that were recently insulated with cellulose insulation which had been treated with inorganic ammonium salts. Following these investigations the French Authorities concluded that the source of the complaints was ammonia coming from the recently installed cellulose insulation material treated with inorganic ammonium salts.

Exposed people from the sites insulated with cellulose insulation treated with inorganic ammonium salts were examined in two studies (CCTV[3], 2013a,b, Annex 3, 4). The French poison control centres (CCTV) found respectively 15 (of 19 exposed) people and 22 (of 43 exposed) people had complaints (mainly mild or moderate symptoms of irritation of mucous membranes). The residents complained about irritation of the eyes, cough, nasal irritation, irritation of the pharynx, other respiratory signs (difficulty in breathing, bronchiolitis) and bronchospasm (listed in almost the same order of frequency in both studies).

CCTV considered in the majority of cases the causality of ammonia as likely to be caused by the cellulose insulation material that was treated with inorganic ammonium salts. In some cases symptoms were reported to start 2-3 days after installation and persisted for up to 16 days after cessation of exposure. Symptoms disappeared following removal of the insulation material.

The dossier also reported that the ECIMA[4] recorded 115 reports of complaints in France while many complaints were made on Internet forums. As the information given on the nature of the symptoms (either smell or/and irritation) and the likelihood of a link was not assessed, these records do not add to the overall evidence of residents suffering from irritation symptoms. The dossier submitter proposed that this information may support the

[3] French committee of toxic vigilance.
[4] European Cellulose Insulation Manufacturers Association.

number of cases being underestimated.

The toxicity of gaseous ammonia related to the observed clinical signs was characterised as irritation to the respiratory tract and eyes following acute and sub-acute inhalation exposure (for days or some weeks). Summaries of other hazards resulting from systemically available ammonia and from dermal and oral exposure are reported in the dossier. They were not considered for the risk assessment of this proposal as other hazards do not correspond to the local irritation effects on the mucous membranes. In this opinion the description of the hazards is targeted to the endpoint 'irritation to the respiratory tract (and eyes)'.

There is no evidence from the observed occupational cases and from those residents making complaints, and living in houses that were recently insulated with cellulose insulation, that ammonia emissions were related to other health effects including de-novo generation of asthma. Asthma-like symptoms were observed in two out of five workers of a plumping company who experienced irritation symptoms after cellulose wadding insulation had been laid down at the construction sites (Annex 4 of the Background Document). The follow-up visit to a physician did not confirm that the asthma was related to the wadding material (negative challenge test) in one case, and in the other case the symptoms disappeared in a few weeks (which contradicts the diagnosis of asthma). Other studies mentioned in the dossier that referred to case reports of occupational asthma were of limited validity as individuals were not exclusively exposed to ammonia, provocation testing (confirming that ammonia was the monocause) by a physician is lacking (Lee et al., 1993, Weir et al. 1989), and in the study of Ballal et al. 1998, a higher risk of asthma was reported for smokers only.

The odour of ammonia gas is pungent. Exposed people may feel affected by the unpleasant odour (smell was recorded in CCTV 2013a,b), but the odour alone does not cause any harm. RAC shares the view of the dossier submitter that the unpleasant odour of ammonia or the general discomfort from the pungent odour it causes, is not considered for the hazard assessment.

For the irritation effects on the respiratory tract and eyes, the dossier proposes a LOAEC of 50 ppmV (35 mg/l) using the Verbek et al. study (1977) as a key study. In that study, self-reporting of symptom ratings for the sum of symptom scores were increased and mild eye and throat irritation occurred at 50 ppmV following 30, 60 or 120 min of exposure.

In addition, RAC finds the study of Smeets et al. (2006) informative. It estimated the intranasal lateralization threshold (LT) of ammonia vapour which is an objective measure of sensory irritation. Within a 2-week period the odour threshold and the LT was obtained twice in 24 healthy, non-smoking volunteers using a static and a dynamic test method (airflow 20 l/min). In this study mean LTs for ammonia were found at 31.7 (static) and 60.9 ppmV (dynamic). In the same range Wise et al. 2005 reported LTs of 37-67 ppmV ammonia.
Smeets and co-authors noted that in individuals, some fluctuations in LT (as well as in odour threshold) is reported to occur due to differences in nasal patency, time of day, health conditions. The mean on the results of static and dynamic methods (46.44 ppmV) is similar to the 50 ppmV of the Verbek study.

The summarised data on the dose-response effectiveness of ammonia vapour (Table 6 of the Background Document, on studies evaluated by the Nordic Expert Group (2005) indicated that symptoms of irritation could occur even at lower concentration than 50 ppmV ammonia. Increased ratings for symptom scores and olfactory symptoms at 10-20 ppmV were reported in 33 volunteers. The original publication (No. 80 in the Nordic Expert Group document, which is only an abstract (Hoffmann et al., 2004)) concluded that the ratings were relatively low (without details at 10 and 20 ppmV ammonia). The corresponding full publication of Ihrig et al.(2006), stated that the mean intensity of respiratory and irritative symptoms lies between 'not at all' and 'hardly at all' even at 50 ppmV. Unfortunately the eye irritation reported in 9% of volunteers at 50 ppmV in the abstract was not documented as a separate effect by Ihrig et al. (2006). RAC takes this study as supportive for the LOAEC of 50 ppmV.

Increased average ratings of eye discomfort (burning, irritated or running eyes) were recorded for 12 healthy volunteers exposed to 5 and 25 ppmV during 3 hours of exposure (Sundblad et al., 2004). Three participants experienced secretion from the nose, and two reported increased cough after exposure to 25 ppmV. Sundblad et al. found that significantly higher discomfort of the eyes was already self-reported at 5 ppmV ammonia. These were estimated as an average pre/post exposure increase of 3.6 mm in a 0-100 mm visual analogue scale (VAS). Although the effect was concentration-related (14.8 mm reported at 25 ppmV), the levels of severity gained were minor. Six mm in the self-rating corresponded to 'hardly at all, while 'somewhat' corresponded to 26 mm on the 100 mm VAS scale. Other irritation effects observed at 25 ppmV ammonia were also in this scale. Nose burning, irritation or runny nose reached 15.3 mm and throat or airway discomfort reached 14.2 mm on the VAS scale.

RAC is aware of some degree of variability in the irritation threshold. Based on the available information RAC chose 50 ppmV as a robust LOAEC. This value is mainly based on the Verbek study and the recent studies of Smeets et al. that use the objective lateralization threshold method to estimate the irritation threshold.

Calculation of the DNEL

Based on the LOAEC of 50 ppmV, a short-term DNEL was calculated by the dossier submitter. An assessment factor of 3 was proposed to adjust the LOAEC to a NOAEC and an intraspecies factor of 10 was used to cover differences in susceptibility among individuals in the general public.

RAC considers an assessment factor of 3 as appropriate to adjust for the lack of a NOAEC.

JRC (2005, The INDEX project) referred to a study of Shim and Williams (1986) who observed that 80% of 60 asthmatics claimed about an exacerbation of asthma following exposure to household cleaners containing ammonia.
Among the cases reports (Annex 4 of the Background Document) there was one case of asthma decompensation of a known asthmatic, a 6-year old child. Although other causes were not addressed, the data may provide some indication that there is a potential of a more severe course of the asthmatic symptoms. This case could be related to the observation that known asthmatics are expected to be particularly vulnerable to respiratory irritants. In contrast, the study of Sigurdarson et al. (2004) (cited in Nordic Expert Group, 2005) could not find changes for pulmonary function or bronchial hyper reactivity after metacholine challenge when 6 healthy volunteers and 8 subjects with mild asthma were exposed to 16-25 ppmV ammonia for 30 minutes.

Sensitivity in terms of a response to a lower minimum effect concentration cannot be excluded for asthmatics, as no data is available (to the knowledge of RAC) that establishes a lower LOAEC for ammonia in this group.

Although an exacerbation of symptoms in people with an asthma history cannot be excluded, RAC proposes to apply an assessment factor of 10 (default value for consumers) to sufficiently protect all parts of the population including children, elderly and asthmatics.

Table 1: Short-term DNEL for the general public exposed to gaseous ammonia (for the endpoint 'irritation to the respiratory tract')

LOAEC	Correction for lack of NOAEC AF	Intraspecies differences AF	**DNEL** LOAEC/ (3 x 10)
50 ppmV (35 mg/m³)	3	10	**1.7 ppmV** (1.3 mg/m³)

Conclusion: RAC concluded that the description of the hazards should be targeted to the endpoint 'irritation to the respiratory tract (and eyes)'. RAC has considered the degree of variability in the irritation threshold, and based on the available information RAC has chosen 50 ppmV as a robust LOAEC. RAC concurs with the calculation of a short-term DNEL and considers the assessment factor of 3 as appropriate to adjust the LOAEC to a NOAEC.

Information on emissions and exposures

Seventeen homes insulated with cellulose insulation were tested by the French Authorities, 14 of which made complaints (CETE, 2013). At three of the 14 sites the level of ammonia concentrations from measurements using diffusion tubes (8 h, detection limit (DL) 2.5 ppmV) grossly matched the concentrations from spot measurements (DL 0.25 ppmV). At two of the sites no ammonia was detected and this may or may not be explained by the point in time when the measurements were undertaken.

The ammonia concentrations at eight other sites were ≤ 2 ppmV in the spot measurements (at the attic or the living-area or both) and were negative in the diffusion tube method (which is consistent as it is below the detection limit of the diffusion tube). The highest value measured was 3.1 ppmV. This data (CETE, 2013) is not published.

In addition another set of (spot) measurements from the French committee of toxic vigilance coordination reported from three properties (in 2012) and four properties (in 2013). Ammonia was found at six of the seven properties.

The maximum concentration measured was 9 ppmV (at one property), up to 3 ppmV (at two properties) and below 1 ppmV (at three properties) (CCTV, 2013 1,b).

As all measurements were retrospective, it is unclear what time lag existed between the installation of the insulation and the beginning of the symptoms. RAC considers that as complaints about odours followed rather rapidly after installation of the material and that the values measured by the French Authorities may have underestimated the concentrations in the early phase after installation. This conclusion is also supported by dynamic testing of the cellulose insulation material, under controlled conditions using the test chamber method according to the principles of test method EN ISO 16000 Standards for the characterisation of volatile pollutant emissions from building products (series of reference standards for the regulatory labelling of volatile organic compounds [COV]) and in particular, the emission test chamber method: *EN ISO 16000-9: Indoor air – Part 9: Determination of the emission of volatile organic compounds from building products and furnishing – Emission test chamber method (AFNOR,2006)*that was undertaken by CSTB. Eleven samples, of treated cellulose insulation, were tested in accordance with the test chamber method EN ISO 16000-9. This revealed that under conditions of high relative humidity (>70%) ammonia is emitted from the material but that emissions levels decrease with time. This evidence supports the RAC's conclusion that measured values may have underestimated the ammonia concentrations in the early phase after installation. RAC agrees that the evidence reported in the dossier, linking the complaints of ammonia odours with the cellulose insulation material containing inorganic ammonium salts is sufficient to conclude that the use of inorganic ammonium salts in cellulose insulation was the root cause of the irritative effects on eyes and respiratory tract reported in the complaints.

The key factors that contribute to the release of the ammonium salts from cellulose insulation are

- Relative Humidity (>70%);
- Loading rate (density/thickness) of cellulose insulation used.

The "type/area" of insulation is also important, with cellulose insulation material in the attic emitting more ammonia than cellulose insulation from walls. As a consequence any measures to ensure compliance of attic insulation with the emission limit value should also ensure compliance with wall type insulation.

The alkaline pH and moisture content of the cellulose insulation and of any material that may come into contact with the insulation *in situ* also plays a role in promoting emissions. However, there is insufficient scientific information in the dossier to determine what levels of moisture in the material are critical to this release.

In addition, the dossier submitter tested different types of attic insulation and found there was also a variation of ammonia emissions within different suppliers. However, they were not able to establish the reasons for this and whether it related to the type and concentration of ammonium salt used, moisture content or pH etc.

Another factor that impacts on the level of ammonia in a specific area is a lack of ventilation. The installation of a ventilation system in homes may cause the diffusion of ammonia into the living space (as obvious in one complaint in CCTV, 2013a) instead of limiting the ammonia emissions in the attic space.

The insulation technique also impacts on the ammonia concentration (e.g. the airtightness of the floor, waterproof structural elements that prevent the insulation material from becoming wet following water penetration or condensation). The presence of such techniques as vapour barriers prevents exposure to humidity, while high pH materials will increase the amount of ammonia released into the living space. The dossier submitter however indicated that the cellulose insulation material might become humid after installation and then emit ammonia. It is not currently clear to RAC whether a suitable technique using water proof packaging (of rolls or panels of insulation material) is feasible and available.

As the actual measured data in homes is of very limited use for a number of reasons e.g. the small number of samples taken, the sampling technique & more importantly the timing of the sampling following installation, an assessment of exposure under worst case exposures conditions was provided by the dossier submitter based on test data from the dynamic chamber tests. These tests have demonstrated that emissions, under worst case environmental conditions, will peak and then decrease with time. Eleven samples of cellulose insulation material treated with inorganic ammonium salts in powder form and two samples of bio insulation material treated with liquid inorganic ammonium salts were tested to establish which samples emitted the most ammonia.

The emission results from the bio based insulation showed that this material did not emit ammonia levels of concern. Note: Bio insulation is treated with liquid rather than powder ammonium salts. It is not technically possible to treat cellulose insulation with liquid ammonium salt.

Four of the cellulose insulation samples that emitted the highest amount of ammonia were subsequently tested further in a test chamber that was scaled to represent a standard reference room in accordance with the CEN/TC 16516 standard while the air flow rate from the CEN standard is lower than the value indicated in the REACH guidance[5] RAC considered

[5] REACH Guidance R15 ECOTOCTRA & ConsEXPO 0.6 air exchanges per hour (Bremmer *et al*, 2007).

the use of the CEN reference room parameters acceptable.

Table 2: Emission profile of categories of insulation materials tested in the static test and dynamic test chamber

Insulation Material	Max conc. of NH_3 ppmV emitted (24hr static[6])	Max conc. NH_3 ppmV (Dynamic chamber test[7])
Category 1 Insulation material	**573**	**316**
Category 2 Insulation material	116	57
Category 3 Insulation material	105	85
Category 4 Insulation material	15	6
Bio based insulation	4-5	1.2

The test chamber loading rate of 12 kg per m² equated to the cellulose insulation loading rate in France. This was based on a cellulose insulation thickness of 30 cm. RAC notes that insulation is measured in terms of its 'R' value or 'U' value (W/m^2 K). While both values are a measure of insulation effectiveness, either value can be used and extrapolated to the other. The R value is generally referenced in the USA, while U values are generally referenced in the EU. The lower the U value, the better the insulation material.

The R value for Cellulose Insulation[8] is in the order of 3.2-3.8 per inch thickness, with 12 inches providing approximately an R Value of 38.4-45.6. This equates to a European U value of between 0.145 and 0.12.

Data from the EURIMA[9] indicate U values in the EU range between 0.75 in warmer regions to 0.13 in colder regions. Therefore, RAC considers the loading rate of 30 cm/12 inches to represent the worst case loading conditions in the EU. Information received during the public consultation also indicated a loading rate of 30 cm.

Test Chamber results establish that the main environmental factor affecting the release of ammonia is relative humidity, particularly when the RH increases above 70%. The test chamber results demonstrated that up to 50% RH, the emission rate of ammonia from cellulose insulation is constant, however above 50% RH the emission rate increases exponentially.

EN ISO Standard 16000-9 0.5 air exchanges per hour.
Chartered Institute Building Services Engineers CIBSE Guidance B (ventilation 2004) 3 air exchanges per hour.
[6] Static test is a test undertaken over 24 hours where no air exchange occurs.
[7] Dynamic testing was undertaken over a period of 28 days under ISO Standard conditions 16000-9.
[8] Source: US Department of Energy. http://energy.gov/energysaver/articles/types-insulation
[9] *www.**eurima**.org/**u-values**-in-europe/*

Table 3 outlines the average conc. of ammonia emitted from the least stable category of material (Category 1 insulation material) tested which was determined from the following RH 50, 70 and 90%.

Table 3: The average conc. of ammonia emitted from Category 1 material at 50, 70 and 90% values of Relative Humidity (RH)

RH (%)	NH_3 (ppmV)	G (mg/h)
50	4	0.168
70	50	2.1
90	250	10.5

Table 3 demonstrates a significant variation in ammonia emissions between 70 and 90% RH. RAC concluded, based on the scientific data available, the equivalent worst case RH for the living area would be less than 70% RH. Values above 70% RH in the living area would result in the formation of moulds within the home. Findings of the OQAI report[10] which recorded RH levels in French homes between October to April and May to September during the period 2004/2005 reported a 95%ile RH value of 64.7%, further supports the RAC's conclusion. While RAC agrees that the RH values in the living area would be less than 70%, RAC also agrees that a RH concentration of 90% could be reached under worst case conditions (depending on the weather conditions) at certain times of the day for a number of days during the year, in the attic area.

Using the well mixed room model the distribution of ammonia in the living area was calculated.

Between 20 and 50% RH, the dossier submitter assumed a constant emission rate equal to the emission at 50% RH. For ammonia emissions above 50% RH the dossier submitter took into account experimental data up to 80% RH. Table 3 shows there is a significant variation in ammonia emissions between 70 and 90% RH (50 ppmV to 250 ppmV). This distribution gave a concentration of up of 3.736 ppmV NH3 in the living room when the RH distribution was between 20 and 50%.

When ammonia exposures were calculated for the living areas based on RH values between 50 and 70% RH, the resulting median was estimated to be 7.948 ppmV and 95%ile of 21.38 ppmV.

These estimated exposures also correlate with the measured data, thus confirming that the least stable cellulose treated material found on the French market, exceeded the derived DNEL under expected conditions of relative humidity in the home when it was loaded at a rate to achieve the R value requirements under French building standards.

In the presence of water inorganic ammonium salts dissolve and an equilibrium is formed between the ionised and the unionised forms. Depending on pH and temperature, relatively more ammonia (NH_3) will be formed (e.g., at pH7, 0.4%; at pH 8, 10%; at pH 9, 50%), which can be liberated as a gas.

$$NH4^+ + H_2O \leftrightarrow NH_3 + H_3O^+$$

Emissions of ammonia have occurred after the cellulose insulation was installed. Solid ammonium salts that are used to treat cellulose insulation can release the ammonium ion in wet/humid conditions crucially when the RH is >80% which is close to the breakpoint in

[10] OQAI (2007). Observatory for indoor air quality – National housing campaign: State of the air quality in French housing, Final report, Report DDD/SB-2006-57 (updated May 2007).

humidograms of several inorganic ammonium salts[11,12,13]. Such conditions could be reached at certain parts of the day in the attic space when the external climate is also humid.

In addition, pH is an important factor influencing NH_3 release. The potential for release of the dissolved ammonia gas is largely governed by the alkalinity (pH) of the solution. pH towards higher values (pH 10-12) will result in a significant loss of NH_3. Lime, plaster and cement are all alkaline and can theoretically react when in contact with the ammonium salts in the cellulose insulation. In one residents complaint, the release of ammonia occurred after the laying of a concrete screed, so it is possible that this may have promoted the reaction, while in another residents complaint release is reported to have occurred when the insulation was in contact with Placoplatre® plasterboard partitions.

During the public consulation (and follow up contacts), industry suggested that a derogation should be considered for composite integration/construction panels such as cladding which are intended for outdoor use only. Industry was asked for more information on the composition of these outdoor articles. No additional information was brought to RAC's attention that cellulose insulation (containing ammonium salts) is used in these outdoor applications.

As the risk of exposure to ammonia from cellulose treated with inorganic ammonium salts occurs when ammonia is released into the indoor environment, RAC agrees that insulation articles, such as outdoor cladding/ construction panels, when structurally designed for outdoor exterior use only would not pose a risk to household occupants.

RAC does however agree that loose fill cellulose insulation treated with inorganic ammonium salts and used to insulate the cavity area in external walls or insulation articles such as panels designed to be used to insulate the external wall of a home from the inside could pose an exposure risk. Therefore such products should be covered by the proposed restriction.

Conclusion: RAC notes that the evidence reported in the dossier is sufficient to conclude that the use of inorganic ammonium salts in cellulose insulation was the root cause of the irritative effects on eyes and respiratory tract reported in the complaints. Concerning the key factors that contribute to the release of the ammonium salts from cellulose insulation RAC considers that (i) the loading rate of 30 cm/12 inches to represent the worst case loading conditions in the EU (ii) while RH values in the living area would be less than 70%, RAC also agrees that a RH concentration of 90% could be reached under worst case conditions (depending on the weather conditions) at certain times of the day for a number of days during the year, in the attic area. In addition, while the air flow rate from the CEN standard is lower than the value indicated in the REACH guidance[14] RAC considered the use of the CEN reference room parameters acceptable.

Characterisation of risk(s)

Ammonia concentrations have been estimated using the Well-Mixed Room model based on the data from the chamber tests for the least stable cellulose insulation material tested at levels of relative humidity in the home living area between 70-90% (worst-case approach).

While RAC considers estimated exposures based on RH values above 70% to overestimate the expected ammonia concentrations in the living area, RAC notes that estimated

[11] http://www.atmos-chem-phys.net/6/755/2006/acp-6-755-2006.pdf
[12] https://uwspace.uwaterloo.ca/bitstream/handle/10012/3683/Rocsana%20Pancescu%20Thesis_5_.pdf?sequence=1
[13] https://pubweb.bnl.gov/~xujun/research/98JPCpaper.pdf
[14] REACH Guidance R15 ECOTOCTRA & ConsEXPO 0.6 air exchanges per hour (Bremmer *et al*, 2007).
CEN Standard 16000-9 0.5 air exchanges per hour.
Chartered Institute Building Services Engineers CIBSE Guidance B (ventilation 2004) 3 air exchanges per hour.

emissions based on the least stable material, found on the French market, under conditions of 50% RH in the living area, were 3.736 ppm. In addition, the median value and 95%ile value under worst case RH conditions (50-70%) yielded estimated exposures of 7.948 and 21.38 ppm respectively. All of these values are above the derived DNEL resulting in all RCR's >1 and demonstrating that the risk is not controlled when the least stable material is used.

Table 4. Risk characterisation ratios (RCR) calculated based on emissions using the least stable material found in the French market

Relative Humidity (%)	**Sub acute inhalation DNEL for irritation**	**Living room**	
		Ammonia Concentration (ppmV)	RCR
20-50% RH	1.7 ppmV	3.736	**2.2**
50-70% RH Median		7.948	**4.7**
50-70% RH 95%ile		21.38	**12.6**

- **Which human populations or environmental compartments are at risk?**

The population at risk are the occupants of properties (primarily occupants of homes) that have been insulated with cellulose insulation treated with ammonium salts which emit ammonia after installation. The population at risk includes all groups of the human population including children and elderly people.

- **Evidence that the existing risk management measures and operational conditions implemented and recommended by the manufactures and/or importers are not sufficient**

Data in the dossier accounts for less than 200 complaints out of the estimated 20,000 homes insulated with cellulose insulation treated with inorganic ammonium salts. The number of real incidents and complaints reported in France, which is one of the primary Member State that has used cellulose insulation treated with ammonium salts, is an indication that current operational conditions recommended and implemented by the manufactures and/or importers are not sufficient assuming that co-factors were present that contribute to the release of ammonia.

As a consequence, (as a safeguard measure), the 2013 French order not only prohibits the placing on the market, sale distribution of cellulose insulation treated with ammonia salts but has also required cellulose insulation material to be removed from homes so no further complaints could reasonably be expected for consideration.

- **Evidence that the existing regulatory risk management instruments are not sufficient**

Construction Products (CP's) are currently regulated under Construction Product Regulations No: 305/2011(CPR). RAC has noted whilst there are currently no limitations on emissions (including ammonia) from CP's in the CP Regulations, where Article 58 deals with complying construction products which nevertheless present a risk to health and safety. "*Where, having performed an evaluation pursuant to Article 56(1), a Member State finds that, although a construction product is in compliance with this Regulation, it* ***presents a risk*** *for the fulfilment of the basic requirements for construction works,* ***to the health or safety of***

persons *or to other aspects of public interest protection, it shall require the relevant economic operator to take all appropriate measures to ensure that the construction product concerned, when placed on the market, no longer presents that risk, to withdraw the construction product from the market or to recall it within a reasonable period, commensurate with the nature of the risk, which it may prescribe."*

Comments received from the Forum indicated from an enforcement perspective that the restriction could be better regulated under the European construction product legislation. The construction products legislation has a requirement for compliant construction products to be CE marked, making the checking of compliance easier. In addition, one Member State comments clearly supported the regulation of this issue under the Construction Products Regulations. The Commission, however, has indicated that the Construction Products Regulation serves to harmonise the test methods performed on construction products, and ensure that the product performances reached and declared by manufacturers are calculated using the same test methods. The prohibition or limitation of certain components in construction products is not the main aim of the Construction Products Regulations but left to be regulated by Member States or other EU legislation (such as REACH). Therefore, the current regulatory risk management instruments are not sufficient.

Conclusion: RAC considers that estimated exposures based on RH values above 70% may overestimate the expected ammonia concentrations in the living area. However, RAC notes that (i) estimated emissions based on the least stable material found on the French market under conditions of 50% RH in the living area, were 3.736 ppm and (ii) the median value and 95%ile value under worst case RH conditions (50-70%) yielded estimated exposures of 7.948 and 21.38 ppm respectively. Since these values are above the derived DNEL resulting in all RCR's >1 it is properly demonstrated that the risk is not sufficiently controlled when the least stable material is used. In addition, RAC concluded that the current regulatory risk management instruments are not sufficient to control the risks.

JUSTIFICATION THAT ACTION IS REQUIRED ON AN EU WIDE BASIS

This is a REACH Annex XVII restriction proposal by France targeted at the use of inorganic ammonium salts (which is used in powder form) as a flame retardant in cellulose insulation. Up until 2011 in France, boric acid was added to cellulose insulation as a flame retardant. However, following the classification of Boric Acid as toxic to reproduction Category 1B under the CLP legislation, the French Authority (CCFAT/DHUP Direction of habitat, urban planning and landscapes) no longer issued technical approvals for the use of boric salts in insulation materials. This resulted in the cellulose insulation sector changing to inorganic ammonium salts (in powder form) as the alternative flame retardant.

Following complaints from occupants and concerns surrounding the release of ammonia from cellulose insulation, the French Authorities introduced urgent national measures prohibiting the placing on the market, import, sale and distribution and manufacture of cellulose insulation containing inorganic ammonium salts as additives. Following consultation with the Commission it was confirmed the issue was not currently regulated under current EU Legislation (CPR). Therefore, action was necessary to address the risks.

As there is no significant import of insulation material, insulation materials are mainly produced in the EU Member States. The dossier identified six producers outside France producing cellulose insulation with ammonium salts. Although no cases were reported from other countries, RAC considers it likely that complaints could arise in other Member States as significant concentrations of ammonia are expected under comparable application conditions using insulation material containing inorganic ammonia salts.

Consideration of the hazards associated with alternatives in the justification for action

While information received[15] from other Member States across the EU indicates the primary flame retardant product used in cellulose insulation is Boric Acid/boron compounds and not inorganic ammonium salts, inorganic ammonium salts are currently used in 5% of the cellulose insulation products in the EU (Source ECIA).

The public consultation revealed some information that ammonium polyphosphates may have a low potential to generate ammonia. However no evidence was provided on the amount of ammonia released from cellulose insulation treated with polyphosphates.

All (4) borate substances with harmonised classification as toxic to reproduction (1B) [boric acid, disodium tetra borates, tetra boron disodium heptaoxide hydrate, diboron trioxide] are currently listed in the Candidate List of SVHC, which is the first step of the authorisation risk management process. Currently they are included in the ECHA's draft 6th Annex XIV recommendation (for inclusion to the Authorisation List). The ECHA's final 6th Annex XIV recommendation to the Commission is expected in Summer 2015.

Specific concentration limits between 3% and 5.5% apply for the 4 borate substances based on Annex VI of the CLP Regulation.

Two additional borate substances [disodium octaborates] have been proposed by the NL for harmonised classification as Repr.1B. The proposed classification at a general concentration limit of 0.3% was adopted by RAC and is scheduled for inclusion in the 9th Draft ATP of CLP, to be sent to the Commission by January 2015 (for final decision).

The dossier submitter indicated (according to the information on ECHA's website) that there are hundreds of substances containing boron. RAC has not been provided with any information that would indicate which non-harmonised (non-classified as CMR) borate substances can be used as alternatives. If the 4 boron compounds are listed in Annex XIV of REACH, this will likely result to further research on the stabilisation of inorganic ammonium salts (or on other non-hazardous boron compounds) as suitable alternatives.

JUSTIFICATION THAT THE SUGGESTED RESTRICTION IS THE MOST APPROPRIATE EU WIDE MEASURE

RAC has noted the comments of MS's, the Forum and the Commission on the CPR Regulations. RAC agrees that a restriction under REACH would also achieve the desired effect and notes that currently under Annex XVII to REACH (Entry 47), cement (a key material used in construction products) is already regulated under REACH.

Effectiveness in reducing the identified risks

The use of cellulose insulation treated with ammonium salts can be permitted provided the cellulose material does not emit ammonia in concentrations greater than 3 ppmV when tested under the specific conditions to be agreed by CEN.

RAC considers the proposed limit to be sufficiently protective because exposure estimates undertaken using the well mixed room model and the data from the most stable material tested (emission profile as set out in Table 5) resulted in estimated 95%ile ammonia emissions of 0.5 ppmV under RH levels <50% and 0.8 ppmV under RH conditions between 50-70%. These estimated emission values are all below the derived DNEL demonstrating

[15] Only six of the 40 manufacturers of cellulose insulation material inside the EU use inorganic ammonium salts as flame retardants.

the risk is controlled when the material emits ammonia less than 3ppmV. The RCR was 0.5 at the 95%ile of 0.8 ppmV.

Table 5: The average conc. of ammonia emitted from the most stable cellulose material tested[16] at 50, 70 and 90% values of Relative Humidity (RH)

RH (%)	NH_3 (ppmV)	G (mg/h)
50	0.4	0.02
70	0.7	0.03
90	0.9	0.04

Tests were also undertaken to simulate conditions of migration of ammonia emissions from the attic to the living area (two chamber tests) using the least stable insulation material found. The results of the testing showed that concentrations in the living area chamber section of the two chambers were 80% of the emission concentrations in the attic area of the chamber tests. However, when these figures were adjusted for air flow, the corresponding concentrations in the living area were in the order of a twofold difference. Based on an emission profile of 3 pmmV at 90% RH, a concentration of 1.5 ppmV would be expected in the living area under worst case conditions.

Practicality, incl. enforceability

RAC considers that overall, the proposed restriction is a measured response to the situation that arose in France, as it prohibits the use of ammonium salts in cellulose (if the emission rate in standardised testing exceeds 3 ppmV) until such time as industry has undertaken research on the stabilisation of inorganic ammonium salts to achieve the proposed standard of 3 ppmV.

This is an important aspect of the proposal from the viewpoint that certain inorganic ammonium salts appear to be viable alternatives for treating cellulose insulation to the boron compounds which are included on Annex VI of the CLP regulation with a classification of toxic to reproduction 1B. While flexibility is afforded to industry to pursue research on inorganic ammonium salts, the proposal is clear that inorganic ammonium salts cannot be used to treat cellulose insulation unless they are able to achieve the limit of 3 ppmV in any one day when tested under worst case conditions over a period of 14 days. This emission level is the limit below which occupants will be protected.

While RAC has no quantitifiable data it considers that articles manufactured solely for the purpose of exterior external use would not be of concern.

Standard testing of the insulation material should demonstrate that the concentration of ammonia does not exceed 3 ppmV in any 24 hour period over a 14 day test duration when tested under conditions of 90% relative humidity. The standard room parameters should be as specified in the test methods of Technical Specification CEN/TS 16516. The CEN method needs some adaptations. CEN/TS 16516 defines a testing method for volatile organic compound emissions and it is based on ISO 16000 standard series. It has been clarified by the Commission in their consultations with CEN experts that CEN/TS 16516 could, in theory, be used for testing inorganic compounds. However, the conditions of the test chamber would need to be re-defined for ammonia. The measurement of released ammonia can be undertaken by ion chromatography following entrapment in an acid solution. As the release factor of ammonia is linked to the relative humidity and the loading in the test chamber, some harmonised conditions (reflecting the different standards for insulation in different regions/MS) would be needed on the loading factor for the panels/material.

[16] Exposure levels in home were estimated using the well mixed room model and the results of the most stable material to determine if compliance could be achieved.

RAC agrees with the Forum's view that those placing cellulose insulation on the EU market are responsible for demonstrating compliance with the above standard.

Manufacturers are responsible for testing the mixtures and articles placed on the market. However, builders and installers will need to follow installation instructions to prevent the release of ammonia in service life. Conditions of use should be provided by the manufacturer or importer placing the mixtures and articles on the market.

RAC notes that in order to explore whether an amendment to the standard is required or whether a Technical Report/Technical Specification would be sufficient to determine compliance, the establishment of an activity, e.g. a. working group by CEN could be beneficial.

In the absence of an amended CEN method, RAC agrees with the Forum that it may not be possible at this point in time to list an appropriate reference as a testing method in the proposed entry to Annex XVII.

RAC recommends that the Commission considers whether the Annex XVII entry can stipulate the requirement for the manufacturer to include documentation and labelling as relevant to the technical specification for the final conditions of use, in order to ensure compliance with this maximum allowable emission limit of 3 ppmV. Failure by builders and installers of insulation to comply with the conditions of use would then be considered not to comply with this restriction entry.

Monitorability

The Forum expressed their concerns with respect to the costs to enforcement authorities having to undertake such complex chamber testing. In order to address these concerns a draft legal text has been suggested to make provision for (1) those actors placing the cellulose insulation on the EU market would be responsible for undertaking the testing to demonstrate compliance and for providing such test results to the relevant authorities, and (2) that the technical specification documentation and any packaging of the corresponding cellulose insulation material should clearly indicate the final conditions of use for mixtures and articles. This would mean that enforcing authorities could take action, as relevant, against both the manufacturer if the product is non-compliant and against the installer if it is not installed as per manufacturer's recommendations.

BASIS FOR THE OPINION

The Background Document, provided as a supportive document, gives the detailed grounds for the opinion.

Basis for the opinion of RAC

The main changes introduced in the restriction as suggested in this opinion compared to the restrictions proposed in the Annex XV restriction dossier submitted by France are: a) provisions on the required technical specifications and b) a derogation for mixures of cellulose containing ammonium salts that will not have to comply to the emission limit, if used to produce panels that have been tested and found to comply. The basis for these changes was the information received during the public consultation and the advice of the Forum for Exchange of Information on Enforcement.

References (additional)

Ihrig A, Hoffmann, J, Triebig (2006) Examination of the influence of personal traits and habituation on the reporting of complaints at experimental exposure to ammonia. Int Arch Occup Environ Health 79:332-338

Sigurdason ST, O'Shaughnessy PT, Watt JA, Kline JN (2004) Experimental human exposure to inhaled grain dust and ammonia: Towards a model of concentrated animal feeding operations. Am J Ind Med 46:345-348

Wise PM, Canty TM, Wysocki CJ (2005) Temporal Integration of nasal irritation from ammonia at threshold and supra-threshold levels. Tox Sciences 87:223-231