Bundesanstalt für Materialforschung und -prüfung

EN

Concept Additional Tests (CAT):

Concept for Assessment of Safe Life Time of Composite Pressure Receptacles by Additional Tests

For the purpose of

re-evaluation of life-time of UN-Composite-Cylinders in the meaning of a service life test programme after 15 years according to 6.2.2.1.1 Note 2*

and safety assessment prior to determination of retest periods for composite pressure receptacles according to P 200 (2)d Note and P200 (9)*

by the competent authority.

This English version is for informative purposes. In case of differing meaning of the German and the English versions the German version is valid.

The procedure described in the following serves BAM to ascertain the safety of pressure receptacles made from composite materials independent of the mode of transport.

Particular effort is devoted to recognizing the variety of design variables and the complex properties of pressure receptacles designed and tested in accordance with standards. At the same time the specific behaviour with respect to life time assessment and periodic inspection of composite pressure receptacles is considered.

**reference to the UN-Model Regulations with relevant chapters of international agreements as IMDG-Code, IATA-TI, RID/ADR*

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Annex:

Technical Annex Slow Burst Test (SBT):
Test Procedure „Slow Burst Test“ from the 26th of September 2014

Technical Annex Load Cycle Test (SBT):
Test Procedure „Hydraulic Load Cycle Test” from the 26th of September 2014

Technical Annex Statistical Assessment Samples (SAS):
Procedure “Statistical Assessment of Sample Test Results” from the 26th Sept. 2014

The Concept Additional Tests (CAT) described in the following and its annexes replace all preceding versions of the Concept Additional Tests since 31st March 2012 including version 2.1 EN from Nov. 18th 2013 and its technical annexes SBT and LCT.

CAT 1 Introduction

The safety assessment of a pressure receptacle made from fibre reinforced plastic, called composite material or „composites", requires the ascertainment of its current strength properties: The strength in new condition or also the residual strength after years in service.

The protection goal is to prevent harm of operators or unintended bystanders. If a population of composite pressure receptacles shows higher survival probabilities than required in CAT 2, the protection goal is deemed to be met. A population of pressure receptacles includes all items of the same design type with identical production and material. Comparable service loads are additional criteria.

Composite pressure receptacles from mass production can be assessed in line with the procedure in CAT 3 employing samples compiled according to CAT 4. These samples can be assessed concerning their burst strength as described in CAT 5 and their cycle strength as described in CAT 6. For this, the strength of each specimen of a sample has to be determined employing the respective procedure[1] repeated identically until failure. The procedures are described in technical annexes "SBT" and "LCT".

The statistical strength(s) distribution of a population of the assessed design type has/have to be derived from the sample test results in a way permitting determination of the survival probability at the highest assumable level of normal operational loads. The corresponding procedures for this are described in the annex "SAS" referenced in CAT 5 and CAT 6. The derived survival probability permits an estimation of the behaviour of a population of pressure receptacles of the same design type and load history. The term "load history" here includes age and application in the meaning of for example "new from production" or "after 15 years of service in a hydrogen battery vehicle" and so on.
Further aspects of design type or sample behaviour are cycle fatigue sensitivity as described in CAT 7 and leak-before-break behaviour as described in CAT 8.

Degradation of properties during service in most cases causes a reduction of safety. For this reason, a population of pressure receptacles can only be considered safe as long as survival probabilities according to CAT 9 can be expected at least as high as demanded in CAT 2.

Results of these additional destructive tests according to this concept and the subsequent comparison of new specimens and such aged by service permit a deeper insight into the safety properties of design types of composite pressure receptacles. This additional knowledge is considered mandatory to assess the design type specific degradation behaviour of composite pressure receptacles. Such an assessment is a precondition for monitoring the residual lifetime of composite receptacles in a well-founded way as explicitly demanded for UN composite receptacles in Note 2 of the UN-Model Regulations 6.2.2.1.1.

But also outside of harmonized regulations this kind of safety assessment is necessary until sufficient non-destructive testing methods are available. It helps to avoid statistically critical safety conditions of a population of pressure receptacles in service. This is particularly relevant if extended retest periods are aspired.

The confidence in a statistical assessment of populations, as described here, requires appropriate pre-fill inspection for surveillance of damages that may increase degradation processes. Pre-fill inspection shall include external visual inspection before filling and an internal inspection as appropriate for the combination of gas and liner material.

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[1] *Annotation: For the current absence of non-destructive test methods with sufficient reliability.*

CAT 2 Protection Goal and Safety Level

The protection goal deems to be met if the population of a pressure receptacle fulfils the required safety level expressed by survival rate SR or failure rate FR. In absence of legal requests concerning minimum reliability the values shown in Table CAT-FR for sudden rupture are intended for provisory use.

In the case that a pressure receptacle design demonstrates load cycle fatigue sensitivity in accordance to CAT 7, a higher failure rate may be accepted. Condition for this is the demonstration of the leak before burst property (LBB) in accordance with CAT 8.

The table CAT-FR shows maximum failure rates FR for a better reading:

$$SR = 1 - FR \qquad \text{Eq. CAT-1}$$

SR has to be interpreted as reliability for pressure receptacles at any time during service life. If a population of a pressure receptacle of this design type with comparable service history provides these or higher survival probabilities, the protection goal is deemed to be met.

Table CAT-FR: Required maximum failure rates or minimum survival rates as safety criterion for residual strength during service life until end of use

failure mode; gas property / **pV**: pressure-volume-product **(PH·Vol$_{water}$)** in [bars litres]**	leak before burst (LBB) (fail-safe design)		sudden rupture / burst
	inert gas	toxic gas	(independent)
	maximum failure rate of a population of pressure receptacles $SR_{min} = 1 - FR_{max}$ during life until end of service life (EoL)		
pV ≤ 30 000 bar L (= 3 000 MPa L)	$FR_{max} = 10^{-4}$ $SR_{min} = 99.99\%$	(see right⇨ "sudden rupture")	$FR_{max} = 10^{-6}$ $SR_{min} = 99.9999\%$
pV > 30 000 bar L (= 3 000 MPa L)	$FR_{max} < \frac{3}{pV}$ (⇨ $SR_{min} > 99.99\%$)	(see right ⇨ "sudden rupture")	$FR_{max} < \frac{1}{100} \frac{3}{pV}$ (⇨ $SR_{min} > 99.9999\%$)

*** according to the marking on the pressure receptacle*

Note 1: *The reference values of this table and the method for considering the consequences of a failure are under discussion and may be applied different.* [2]

[2] *Annotation for Table CAT-FR:*

1. *The demanded values are based on a rough risk assessment according to failure consequences. This consequence related criterion is built on the pressure-volume-product, the behaviour of the pressure receptacle during failure and properties of the contained gas as e. g. toxicity.*
2. *The pressure-volume-product is a fundamental dimension for the amount of stored energy. This includes the potentially released gas available for a chemical reaction as well as for the physical reaction, which results from the contained energy from gas compression and from elastic expansion of the shell of the pressure receptacle.*
3. *It is assumed that a burst test will lead to a sudden rupture.*
4. *During the assessment of load cycle tests the criterion of leak before burst can be considered additionally.*

Note 2: *Since properties of a population of cylinders in use are unknown, they have to be estimated by sample properties. Relevant test and evaluation methods are presented and referenced in chapters CAT.5 and CAT.6. For assessment of LBB-CAT 8 shall be followed.*

For flammable or oxidizing gasses, which are not mentioned in table CAT-SR the following rules applies:

In general flammable or oxidizing gasses shall be treated like toxic gases.

There are applications where the consequence of a leakage of flammable or oxidizing gas is comparable to an inert gas. In such cases, the requirements on the pressure receptacles can be equalized between inert and flammable or oxidizing gases. This can, for example, be considered applicable to a pressure receptacle containing flammable gas if it is secured at all times during service that its surrounding is free from ignition sources and properly ventilated.

CAT 3 Applications

The safety assessment procedure described in this concept of additional tests is applicable to composite pressure receptacles of all types and sizes made in large numbers. In general this is assumed for design types up to 450 Litres water volume.

The procedure for the determination of survival probability of a population of pressure receptacles described here shall be used either on a singular, repeated or periodic base. It provides a general assessment of the current safety level . In the case of repetition (in the following called: re-evaluation), this procedure provides a comparing assessment of service safety levels. If the procedure described here is used it shall be used as its whole and cannot used partially.

A comparison of safety levels at different ages describes the in-service degradation of pressure receptacles. Therefore the procedure is applicable to a verification of residual life expectancy as well as to the setting of suited retest periods adapted to the expected life time. Safety is confirmed if the survival rate of the relevant population of a design type is demonstrated for the Begin of Life (BoL) and if possible for the End of Life (EoL) as being higher than the minimum required level.

The maximum service pressure is considered the test pressure PH. If a design type is only used for one dedicated gas, the developed pressure at a settled temperature of 65°C in a maximum filled pressure receptacle (working pressure at 15°C) can be assumed as maximum service pressure (MSP).
If the design type approval is not limited to one specific gas, then the value of PH shall be used as MSP in the following including the performance charts Fig. SAS-1 and SAS-2 in annex "SAS". The pressure level in Fig. SAS-3 and Fig. SAS-4 in annex SAS shall be altered respectively.

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5. *Leak before burst behaviour may be dependent on the aging conditions of the receptacle. For that reason this behaviour has to be re-evaluated during every test, if the reduced minimum survival rate based on leak before burst is employed.*
6. *It is assumed that the leakage of inert gasses has no direct consequences opposite to leakage of toxic gas except loss of functionality. The last is not a subject of this safety assessment.*
7. *For this reason, the consequence of leakage of a cylinder filled with inert gas with low pressure and energy dissipation is considered significantly smaller than a cylinder rupture or a leakage with release of toxic or flammable gas.*
8. *The requirements for flammable or oxidizing gasses are guided by likeliness of ignition depending on the application.*

The procedure of initial testing is shown in the **Figure CAT-1**. The explanation of the different steps and variants of this is given in the following chapters CAT 4 to CAT 8 as mentioned in the flow chart.

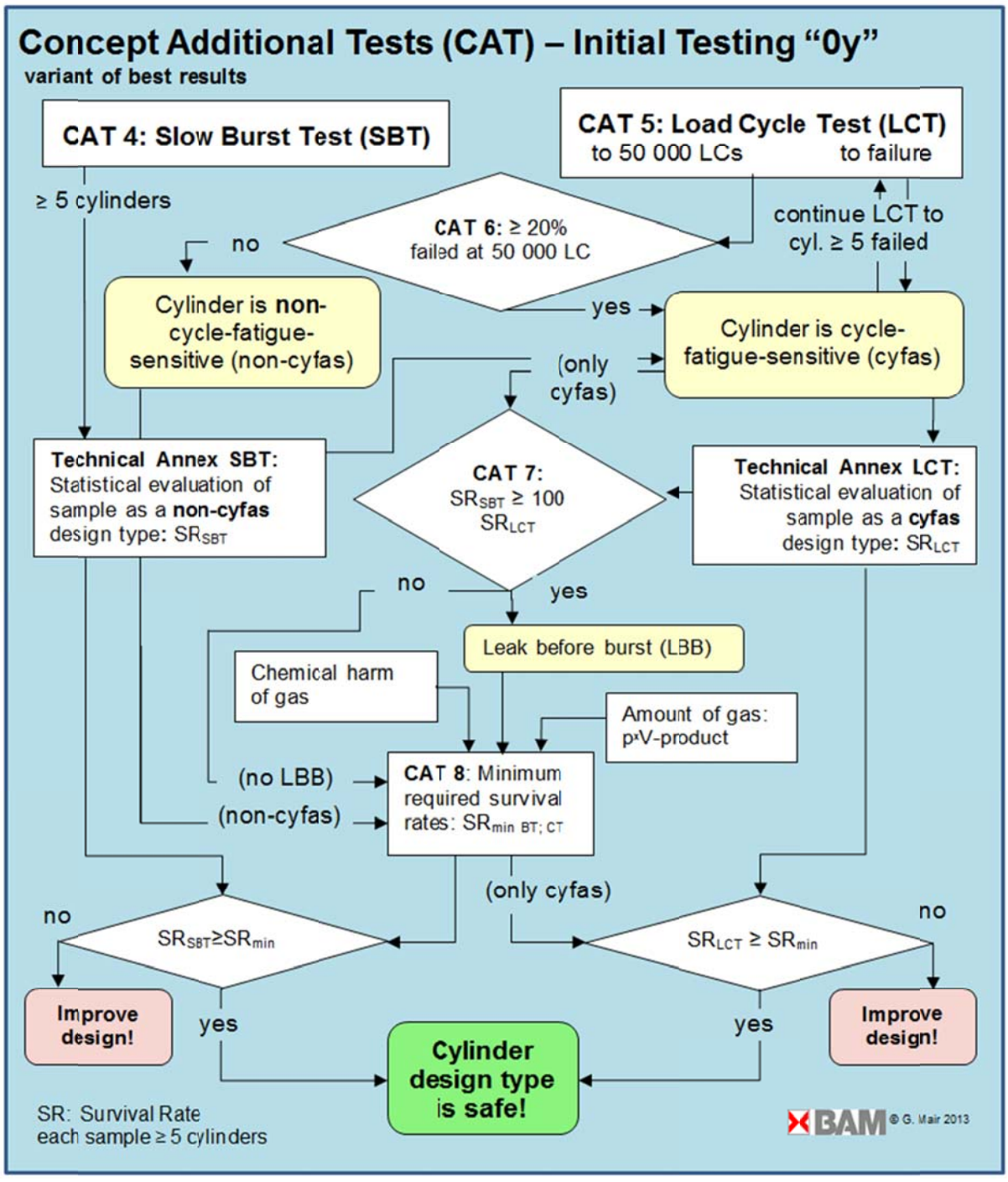

Figure CAT-1 *Procedure of initial testing – intended for application during design type testing.*

Design types not assessable according to this concept of additional tests for the reason of their limited production numbers need to be examined and monitored in an equivalent way approved by the competent authority. The effectiveness of the monitoring methods need to

be proven regarding the early and reliable detection of preliminary signs of a first failure or speeding up of micro cracks. Under this condition those methods can include the permanent online-monitoring based on acoustic emission testing (ISO-short cut: AT) and integrated optical strain sensors.

CAT 4 Composition of Sample

Due to different statistical behaviour of different design types or even design variants each design type variant has to be treated separately.

The competent authority may agree to sampling of different variants in one sample. In this case the manufacturer shall demonstrate sufficient experience concerning production and re-evaluation according to CAT.

Note 3: *Nevertheless, due to statistical effects, this procedure bares the risk that even if the BoL-survival rate (BoL: Begin of Life) of a design is quantified as sufficient, the uncertainty for a similarly sufficient EoL-survival rates (EoL: End of Life) of this design type could be high.*

Each of the test procedures described in the following requires a sample of usually 5[3] specimens. For this purpose the following requirements apply:

(a) Each sample consists of usually 5 specimens with known history (age, gas service(s), intensity of use, climate conditions etc.).

(b) A sample may consist of operated or new specimens, depending on the intention of the test. Only specimens taken from a population with comparable service history shall be compiled to a sample. The service history can only be considered comparable if (d) and (e) are fulfilled at the same time or (f) and (g) respectively.

(c) For a comparison of static load and cyclic load behaviours in accordance with CAT 4 and CAT 5 e. g. for the purpose of leak-before break properties both samples need to have a comparable service history.

(d) If an assessment of new pressure receptacles is intended, the sample must be taken from a pre-production batch or out of as much different batches of a running production as manageable.

(e) In case of new (unused) specimens they shall not be produced more than 3 months before testing.

(f) In case of used specimens taken out of service, dates of manufacturing shall not differ more than 6 months for being considered as being of the same age and composed to one sample either for cycle or burst testing. Also the intensity of their service has to be at least average and traceable.

(g) Before any specimen is subjected to destructive testing it has to pass the non-destructive periodic inspection and all tests as requested for each individual of the population in accordance to the relevant standard.[4]

(h) Samples may consist of specimens of comparable service history from a popula-

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[3] *Annotation: Due to consideration of confidence level there is no minimum samples size. It is recommended to use a sample size of at least 5, Therefore performance charts are based on a sample size of 5. Differing sample sizes may be used but need separate calculation.*

[4] *Annotation: This is to secure the specimens in the sample to be representative for all composite pressure receptacles of this design type in service after periodic retesting.*

tion used at varying operators. In this case every sample has to be increased to at least 8 specimens, while including at least 3 specimens from each operator (1 x 5 ⇨ 2 x 4, 3 x 3 or 4 x 3 etc.).

(i) Results derived from samples taken out of service shall only be applied to the receptacle population of operators who supplied specimens for the sample.

(j) Exceptions from (i) can be considered. For this the manufacturer has to prove that the variety of all service conditions has been covered by specimens supplied by other operators. In this case the sample has to consist of at least 12 specimens from at least 3 operators.

Note 4: *Due to the differences in service the composition of pressure receptacles from different operators may lead to an increased scatter of residual strength.*

CAT 5 Slow Burst Test (Annex "SBT")

Note 5: *Requirements of CAT 2 are graphically displayed in **Fig CAT-2**. There the lines of constant survival rate are presented for the whole population. Since the properties of a whole population are unknown and can just be estimated by sample properties, uncertainty margins must be added as shown in Annex SAS.*

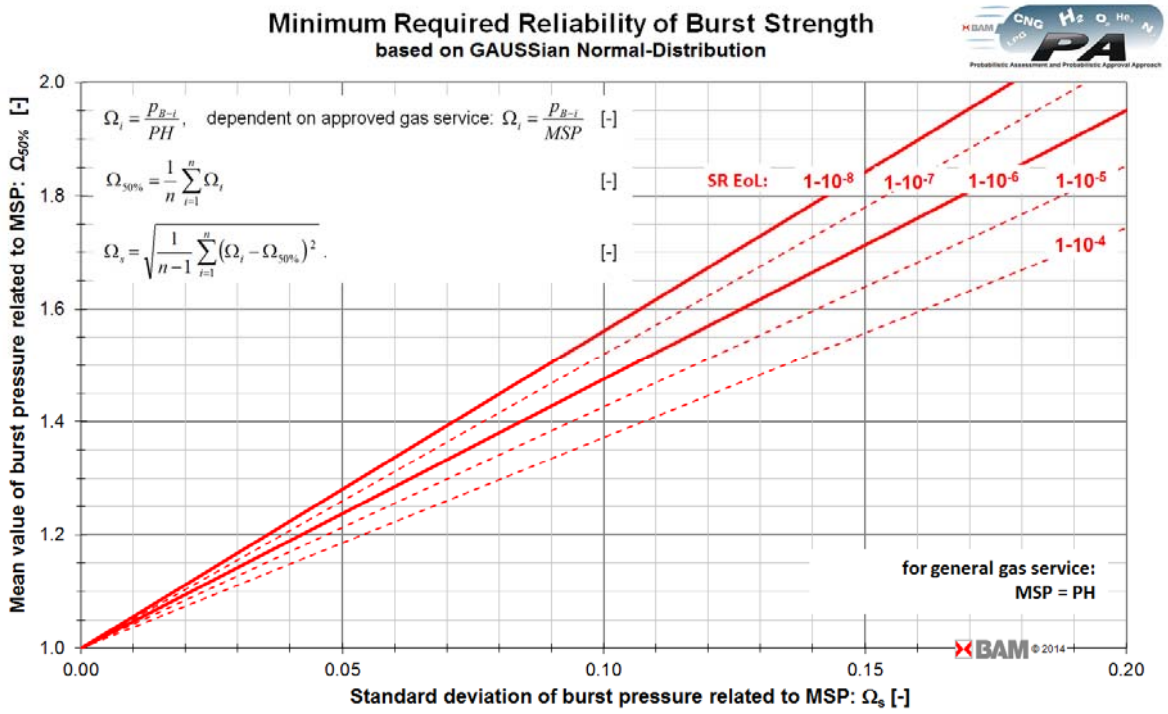

Figure CAT-2 *Performance chart for a graphical assessment of whole population (unlimited sample size) tested by SBT*

First a sample has to be compiled according to CAT 4. The sample shall be subjected to slow burst testing as described in Annex SBT. The test results shall be evaluated graphically or computed according to annex SAS. The resulting sample survival rate based on maximum allowed service pressure (MSP acc. to CAT 3) shall be compared to SR_{min} found in table CAT-FR.

The sample survival rate shall be higher than SR_{min} under the consideration of sample size and a unilateral confidence level of 95%.

If the required survival probability was not achieved, additional pressure receptacles can be tested by enlarging the sample. For this the sample can be expanded arbitrarily in accordance with CAT 4. Independent of sample size, all results shall be directly included in the statistical examination. Deviating from that, single results may be removed from statistics only if it is proven that there was a mal-function in the test equipment or similar and there cannot be any reason for failure in the tested specimen. Such an elimination of test results and each test repetition shall be documented.

If the requirements from table CAT-FR are not fulfilled, the design type shall not be approved for further use. Respectively the use of pressure receptacles of this design type shall end depending on service history.

Note 6: *If this assessment shows insufficient reliability of a sample of cylinders coming from production, there is currently no legal foundation for a refusal of an approval if the mandatory standard is fulfilled. But in this case the manufacturer shall be informed about the detected lack of safety and consider improvement of his design.*

CAT 6 Load Cycle Test at Ambient Temperature (Annex "LCT")

Note 7: ***Fig CAT-3*** *shows the SR_{min}-requirement for a population of composite pressure receptacles as requested by table CAT-FR in a performance chart. This performance chart summarises the mean value and the scatter of load cycle properties and shows relevant lines of constant reliability ("isoasfalia") for the basic population.*

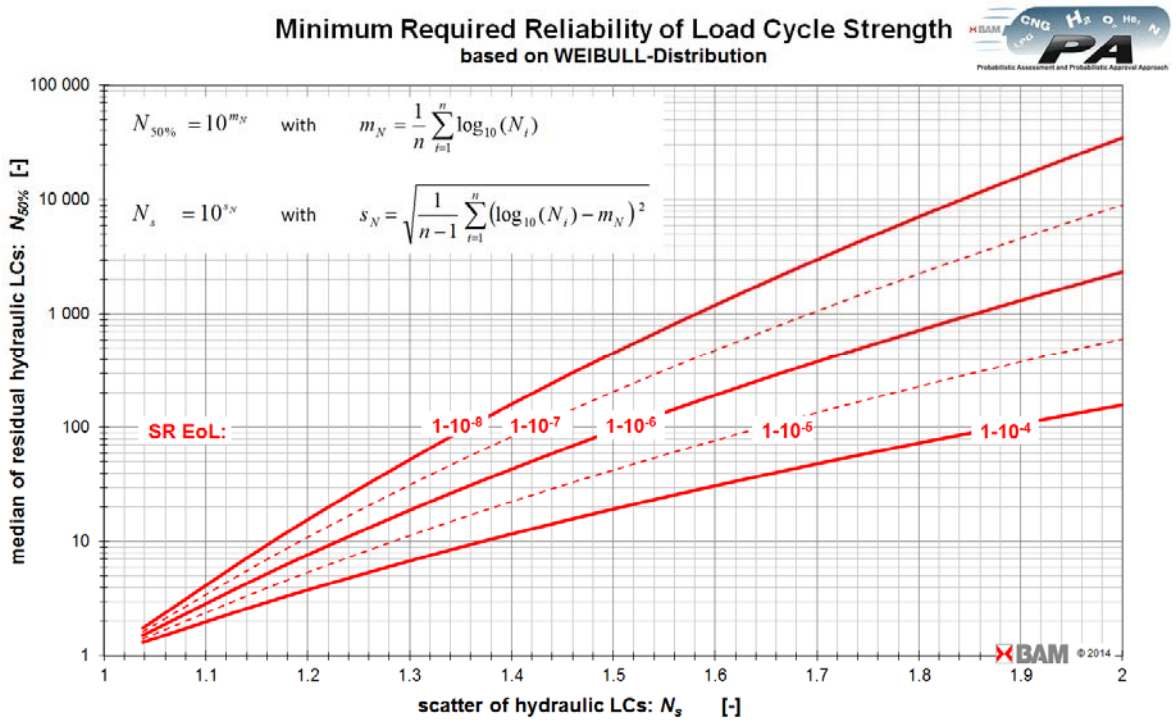

Figure CAT-3 *Performance chart for a graphical assessment of whole population (unlimited sample size) tested by CLT*

After successfully passing burst testing, a second sample compiled according to CAT 4 shall be tested. This sample shall be subjected to load cycle testing at ambient temperature ac-

cording to annex LCT. In case of initial load cycle testing of a design type each cycling of specimens may be aborted at 50 000 load cycles. The conditions for a final stop of cycle testing or for continuation of aborted cycling to failure of the whole sample are dependent from their property with respect to cycle fatigue sensitivity (see CAT 7). The results of each sample completely tested to failure shall be evaluated graphically or calculated according to annex SAS.

The sample survival rate acc. to annex SAS shall be higher than SR_{min} under the consideration of sample size and a unilateral confidence level of 95%.

If the sample shows less than the required survival probability from Table CAT-FR, this sample size may be increased according to preferences or requirements, too. Like demanded in CAT 5 all test results have to be included in the statistical assessment independent of sample size. A test results may only be removed in a case where malfunctioning equipment can be proven and failure caused by specimen performance can be excluded. Such an elimination of test results or test repetition shall be documented.

If the requirements from table CAT-FR and Fig CAT-3 are not fulfilled for the next load cycle, the design type must not be approved; respectively the use of pressure receptacles of this design type shall end depending on service history. See Note 5 above.

CAT 7 Classifications Regarding Cycle Fatigue Sensitivity

If both tests described in CAT 5 and CAT 6 are finished, the design type shall be classified regarding its cycle fatigue sensitivity.

The design type is considered as being **non load-cycle-fatigue-sensitive (non-cyfas)** if none of a sample of at least 5 specimens fail up to 50 000 load cycles. If a specimen of the sample fails, the design type is considered as being **load-cycle-fatigue-sensitive (cyfas)**.

This classification is limited to the known history of population that is represented by the sample(s).

Tests according to annex LCT always have to be continued until failure for samples of load-cycle-sensitive design types. For this reason either all load cycle tests which were cancelled at 50 000 load cycles before classification have to be continued until failure, or a new test sample from the same production batch(es) has to be used for cycle testing until failure.

Load-cycle-sensitivity may depend on the aging condition of the pressure receptacles of a design type. For this reason, the load cycle sensitivity should be reassessed during every re-evaluation of a design type.

CAT 8 Classification of LBB-Behaviour (Annex "SAS")

This is only valid for load cycle sensitive designs in accordance to CAT 7.

The LBB-criterion ("leak-before-break" or better "leak-before-burst") is considered fulfilled for pressure receptacles of a certain design type and service history if the following properties are proven on the basis of survival rate SR or failure rate FR (see annex "SAS"):

a) None of the load cycle tests has caused a rupture before leakage.

b) Results concerning survival rate SR or failure rate FR (FR = 1- SR) are available according to CAT 5 (burst strength) and CAT 6 (cycle strength) for the design type.

c) The design is classified as load-cycle-fatigue-sensitive in line with CAT 7.

d) The failure rate derived from burst tests (CAT 5) is not higher than the failure rate derived from cycle tests (CAT 6) divided by 100: $FR_{SBT} \leq FR_{LCT}/100$.

e) There are no hints for preliminary secondary failure of the composite from the hydraulic or pneumatic cycle tests.

CAT 9 Interpretations of Results (Annex "SAS")

When having completed the procedures of the concept of additional tests as introduced above including both aspects, LCT and SBT, an estimation of reliability of the investigated composite pressure receptacles is available depending on their history.

A well-founded estimation of the design type's degradation is possible, if there are results of the population of pressure receptacles of two or better three different years or manufacturing (i. e. new, after 15 years, after 30 years of service in case of a sufficient original design life) and each strength test (SBT and LCT),. This can result in evidence whether pressure receptacles of this design type may reach the end of their service life in a safe condition.

The requirements for a service life test programme as described in the current Note 2 in 6.2.2.1.1 of UN Model Regulations and the new definition and requirements expected to be part of its 19th revised edition are covered by this procedure. In the case of meeting the safety required in CAT 8, relevant service life limits can be lifted as described in the 19th UN Model regulations 6.2.2.1.1 in conjunction with the Note under 6.2.2.7.4

This procedure, which employs a comparing assessment of samples of different ages from a population, may be considered as current state of science and technology (SST). It cannot be described in a fixed procedure as it is common for the state of the art (SA). For this reason it should be performed by the competent authority on a scientific level until further notice.

CAT 10 Surveillance of Production Scatter (Annex "SAS")

The surveillance of production has two safety related aspects. One is the assessment of each production batch with respect to a manufacturing failure (compliance with the design type). The other is the surveillance of statistical properties of pressure receptacles produced in a dedicated period of time (validity of reliability value). In case of no significant changes of these properties, life time assessment results from pressure receptacles produced in preliminary periods may be taken in consideration and reduce test effort..

Note 8: *This is an aspect of simplification for lifting of the service life limitations. The production batches, which were assessed in accordance with CAT initially, are the base for assessment of following strength behaviour and degradation assessment. This condition is necessary due to relatively short production periods. Only in exceptional cases it is expected to see a production of an unchanged design type for more than 10 years. For this reason in most cases the production will be finished before e. g. the 15 years period of UN-Note 2 becomes relevant.*

CAT 10.1 Production batches

Additional requirement for batch testing of production:

One pressure receptacle per 1000 produced, but at least 5 per annual production have to be taken randomly out of the production for further testing. The test to be used depends on the classification in accordance with CAT 8: In case of a cyfas-design type the LCT shall be used; in case of a non-cyfas design type the SBT shall be used.

Note 9: *It is recommended to check the initial classification according to CAT 8 annually by a complementary sample of cylinders randomly picked out production. This sample should be tested by the test method from the CAT 8 assessment, which is <u>not</u> used for batch testing..*

Each result of an individual test should be higher than the mean value of the initial sample minus two times of the initial scatter value:

Burst test: $p_{batch} > m_{pB} - 2 \cdot s_{pB}$ (see annex SAS 3.1) Eq. CAT-2

Load Cycle test: $N_{batch} > 10^{m_{logN} - 2 \cdot s_{logN}}$ (see annex SAS 3.1) Eq. CAT-3

A) If a test result does not meet this requirement and the test has been carried out in a satisfactory manner, the cause of test failure shall be identified or the batch shall be rendered unserviceable for the intended purpose.

B) If the cause of failure is identified, a sample of three pressure receptacles shall be tested in the same way as the not passed pressure receptacle. If each specimen of this sample meets the requirement, the production batch may be reclaimed.

C) If the cause of failure is identified and one or more of these three pressure receptacles does not meet the relevant Eq. CAT-1 or CAT-2, the whole production batch may be treated with respect to statistical assessment separately in the manner requested for a new design/design variant. In this case, the further production cannot take advantage from the preliminary safety assessments. Otherwise the batch shall be rendered unserviceable for the intended purpose.

CAT 10.2 Annual production

All test results from SBT- and LCT-production batch testing collected during 12 month of production shall be assessed by statistical analysis in accordance with SAS. The result shall demonstrate that each annually produced population of pressure receptacles shows the same minimum level or BoL-survival rate (BoL: Begin of Life) as demonstrated initially.

If test results show a survival rate lower than the initial value of SBT- or LCT-assessment further measures shall be taken:

D) Check significance of deviation between initial sample and batch sample (parameter test in compliance with SAS 3.3). If the batch sample shows a significantly higher scatter or significantly lower mean value, then go ahead with E) or F).

E) Either the conformity with this requirement can be demonstrated by an increase of the number of randomly taken specimens for further data on the relevant annual production;

F) or the composite pressure receptacles from relevant annual production shall be treated separately with respect to statistical assessment as necessary for a new design/design variant and cannot take advantage from the re-evaluation of the cylinders from the first year of production.

CAT 11 Informal Flow Charts

The flow chart Fig CAT-1"Concept Additional Tests (CAT) – Initial Testing "0y" variant of best results" is presented in CAT 2 and shows the order and interaction of decisions as described above.

The **Fig CAT-4** "Concept Additional Tests (CAT) – 15 years Re-Evaluation variant of best results" shows the order of decisions as described above following up the repetition of the procedures shown in Fig CAT-1.

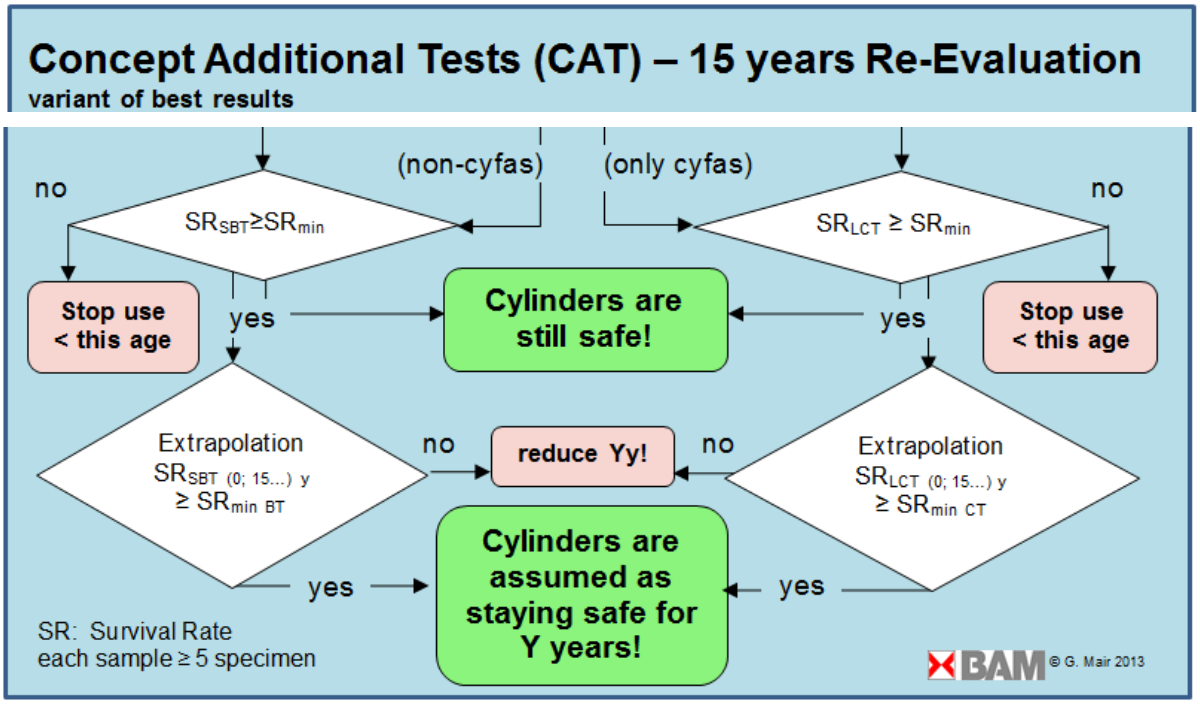

Figure CAT-4 *Additional steps for the comparison of test results for service life testing after the repeated execution of the procedure as shown above for re-evaluation*

A reduced process based on the classification of cycle fatigue sensitivity but without the facilitation in case of LBB-properties is shown in the following flow charts **Fig CAT-5, CAT-6** and **CAT-7.**

These simplifications may be used for the service life testing of design variants derived from design types completely and successfully tested in accordance with CAT.

This reduced program is especially intended for a simplified service life check before the determination of retest periods of more than 5 years in cases where the service life testing in accordance with Note 2 of 6.2.2.1.1 UN-Model Regulations is not required.

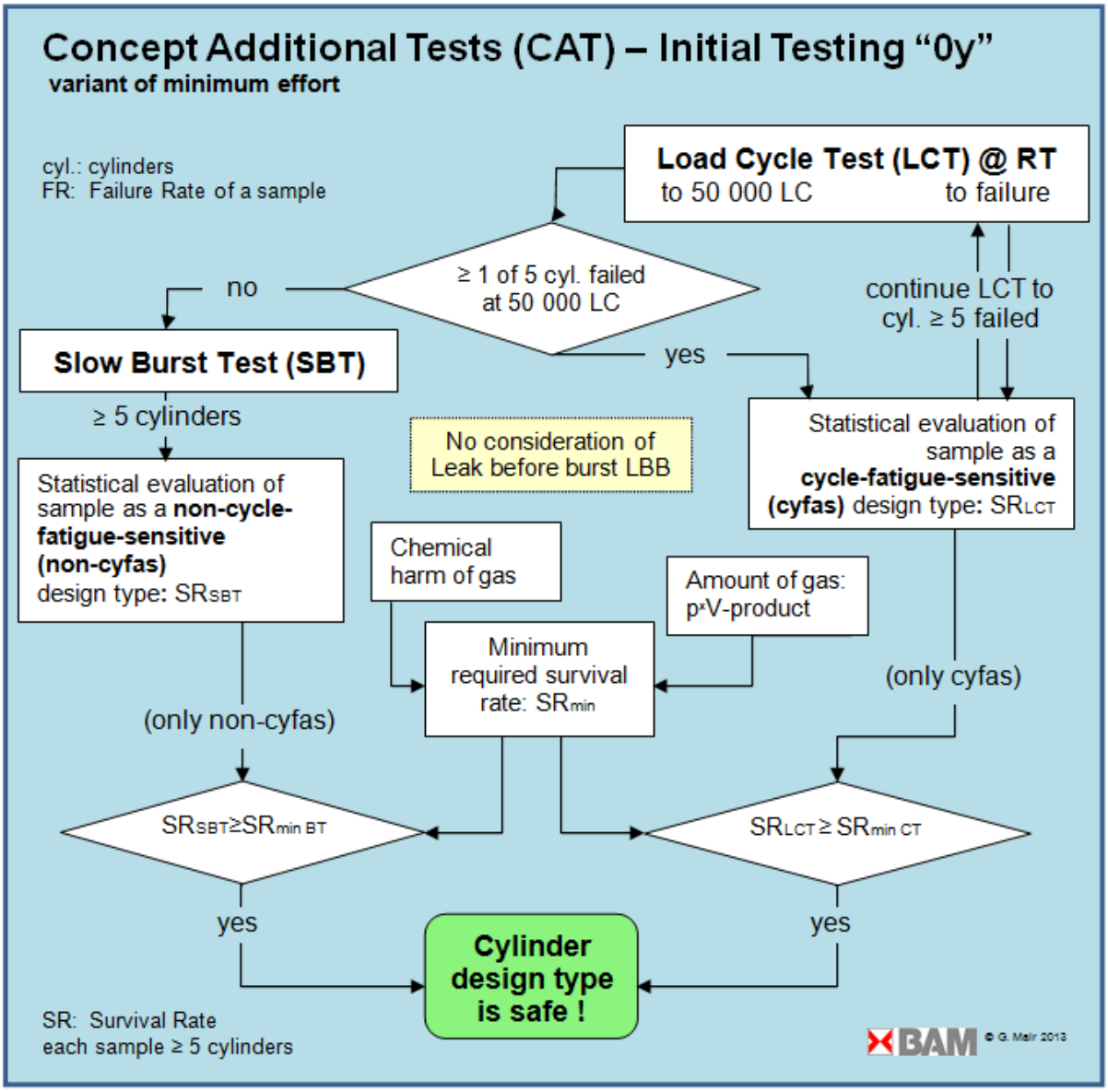

Figure CAT-5 *Reduced procedure of initial testing intended for execution during design type testing.*

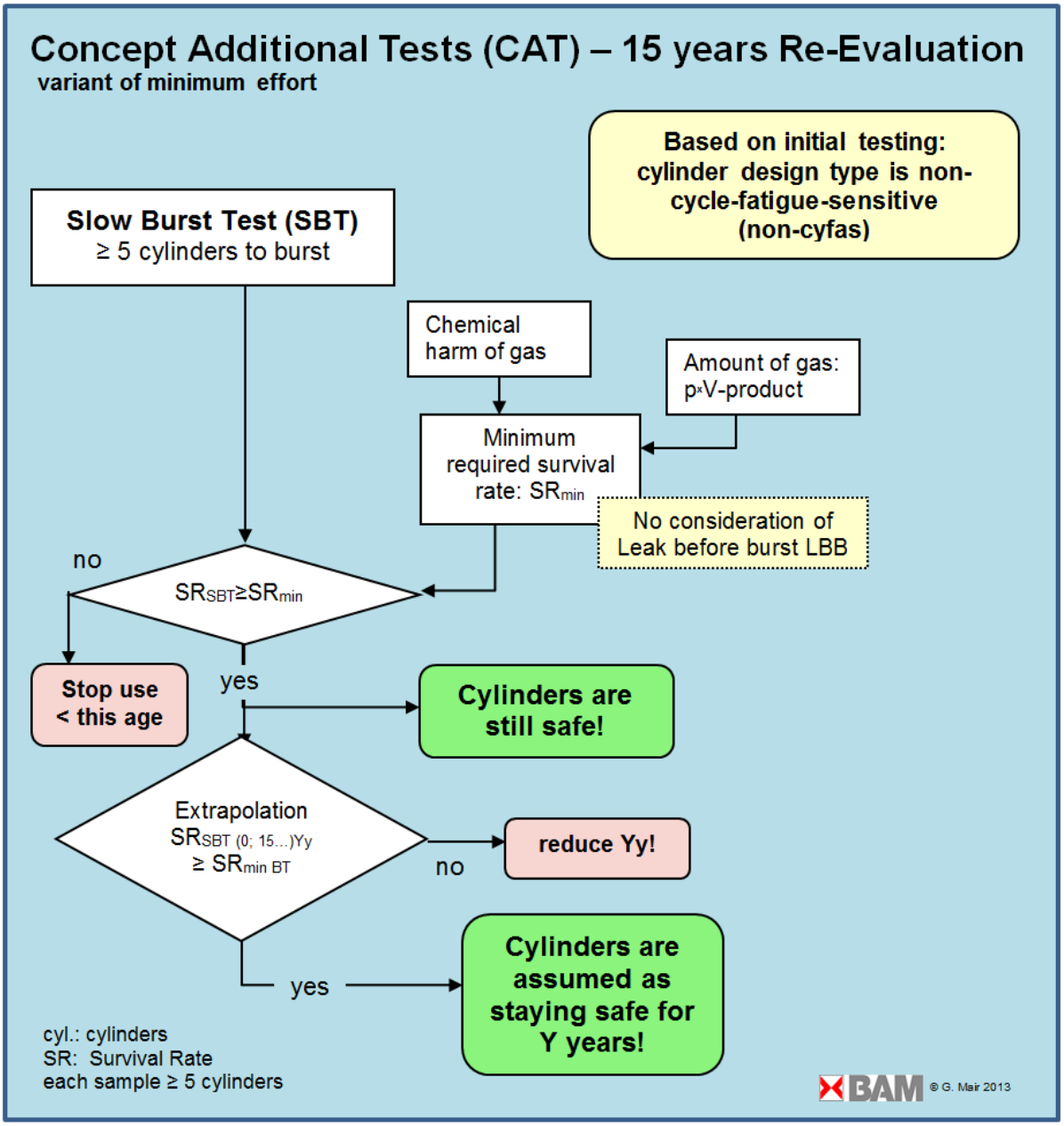

Figure CAT-6 *Reduced procedure for re-evaluation of statistic performance for life time surveillance in case of non-cycle fatigue sensitive designs.*

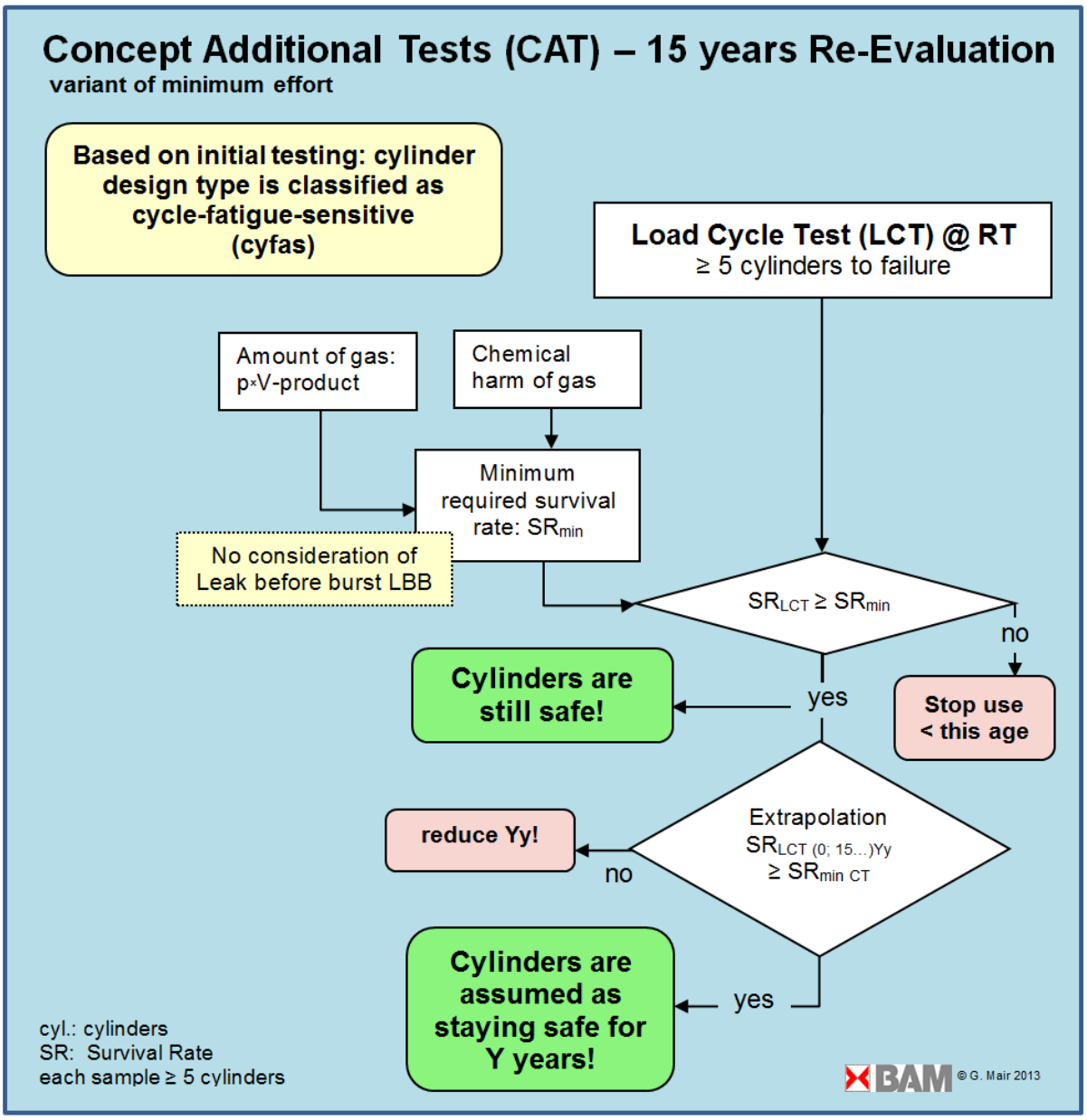

Figure CAT-7 *Reduced procedure for re-evaluation of statistic performance for life time surveillance in case of cycle fatigue sensitive designs.*