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# Characterizing aging of lithium-ion batteries during long-term test campaigns for transport applications

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Abstract—Long-term test campaigns are necessary to understand the aging behavior of lithium-ion batteries in transport applications. During them, a characterization procedure is used to track aging giving both researchers and engineers valuable information. However, no consensus has been found on the procedure to be used. In fact, it is subject to the study under consideration and above all, to time and equipment constraints. Regarding the literature, this work has analyzed and compared noninvasive procedures from past aging test campaigns. Several aspects including duration, data relevance or impact on aging have been discussed. This review aims at helping the reader to choose the most appropriate procedure in line with its application.

#### *Keywords—lithium-ion; aging; characterization; RPT; capacity test; pulse power test; OCV test.*

#### I. INTRODUCTION

During operation and rest phases, lithium-ion batteries age, resulting in a decrease in their storable energy and an increase in their internal resistance [1]. It is therefore necessary to understand the phenomena responsible for aging to optimize their use. In this perspective, accelerated aging tests in laboratory conditions are an efficient way to study the degradations in a reduced test duration [2], [3]. The cells are stressed with specific duty cycles and their aging performances are regularly measured to observe their evolution over time. It allows to study the degradation behavior of the batteries as well as modelling it. For this purpose, an accurate aging characterization procedure also called reference performance test (RPT) is defined. During the RPT, three parameters are usually determined: cell capacity, internal resistance and open-circuit-voltage (OCV) profile. The former two are used to quantify the capacity and power fading, while the last one has been successfully used for several years to synthesize the degradation modes [4].

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In the literature, Mulder et al. [5] reviewed the characterization standards and compared the results obtained by implementing them. Afterward, Barai et al. [6] presented a comprehensive and critical review of the existing methods for cell characterization. Both contributions can help the reader to build its own RPT according to the study under consideration and constraints, such as time and equipment availability. Thus, it can be observed that the RPT used in actual aging studies deviate from each other and standards. The aim of this work is to investigate and compare the different RPT used in aging studies from the literature and see how they were implemented according to their requirements and limitations. It is not an exhaustive review but a critical one based on various real test campaigns. It is restricted to non-invasive tests and is closely linked to transport applications.

In this paper, twelve RPT from literature have been selected and reviewed [7]–[18]. The selection tries to give a broad overview of the work carried to the present day so far. However, it should be noted that not all studies present their protocol in detail and some information may be missing. For this reason, the review may be limited on some aspects. All these studies are summarized in Table 1.

In the next sections, the twelve selected papers are analyzed regarding the three main parameters measured by RPT: capacity, resistance and OCV-profile. Tests used to measure these parameters are given and compared between the different RPT. Then, the content and the timeline of the procedures are investigated. To finish, RPT are studied "as a whole" in the last section, allowing to visualize their strengths and weaknesses depending on the applications considered.

#### II. CAPACITY MEASUREMENT

The capacity measurement is always performed by a capacity test usually composed of a complete charge at constant-current constant-voltage (CC-CV) followed by

Reference	Year	Summary	Application	Ageing test	Cell (chemistry, form, size)
Braco et al. [7]	2020	Accelerated tests of second-life batteries to investigate end of life criteria	EV in first life	1C full charge/discharge cycles	LMO, pouch, 33 Ah
Sarasketa- Zabala <i>et al</i> . [8]	2014	Development of calendar and cycling ageing predictive models	Applications using LFP technology, including transportation	Calendar: different storage conditions. Cycling: different DOD / C-Rate.	LFP, 26650, 2.3 Ah
Grolleau <i>et al.</i> [9]	2016	Accelerated ageing tests combining calendar and cycling	EV HEV	Automotive cycles alternating with resting periods	43 Ah / 26 Ah
Belt <i>et al.</i> [10]	2007	Investigation of the effects of temperature on capacity and power fade	Automotive	Battery cycles under 40°C and 20% DOD	12 Ah
Han <i>et al</i> . [11]	2014	Cycle life comparison of five commercial cells in electric vehicule	EV	Typical EV cycles	NMC+LTO, 20 Ah LFP, 60 Ah LFP, 11 Ah LMO, 10 and 35 Ah
Spagnol <i>et al.</i> [12]	2010	Development of an on-board ageing parameter estimator for life estimations and prognosis	HEV	Synthetic duty cycle profile from a real driving cycle	LFP, 26650, 2.3 Ah
Wang <i>et al.</i> [13]	2011	Study of a specific cell with large number of ageing tests, to build ageing model	Applications using LFP technology, including transportation	Large cycle-test matrix, with different temperatures, DOD, C-rates	LFP, 26650, 2.2 Ah
Bloom <i>et al.</i> [14]	2001	Calendar and cycling aging investigation at different temperatures	Cited by [11] as reference cycles for automotive applications	High temperatures, 2 SOC, and 2 different DOD	NCO, 18650, 0.9 Ah
Gering <i>et al.</i> [15]	2011	Investigation of path dependence in plug-in hybrid vehicle applications	PHEV HEV	Thermal cycling and investigate several typical paths	NMC+LMO, 18650, 1.9 Ah
Li et al. [16]	2018	Accelerated aging tests for SOC estimators	EV	Dynamic current profiles specific from EV applications	NMC, 45 Ah
Martinez- Laserna <i>et al.</i> [17]	2018	Investigation of the degradation behavior of battery in second life	EV in first life Stationnary in second life	Accelerated aging with synthetic stationnary application profiles	NMC, 20 Ah
Baure et Dubarry [18]	2019	Comparison of real driving data to synthetics driving cycles in terms of cell degradations	EV	3 synthetic driving cycles 1 real driving cycle	NCA, 18650, 3.4 Ah

TABLE 1: OVERVIEW OF THE TWELVE AGING TEST CAMPAIGNS SELECTED

a discharge at constant-current (CC). It is performed systematically in each RPT reported in the literature. This test quantifies the evolution of the available amount of charge of the cell. It is therefore an essential indicator of ageing. The capacity is mainly determined in discharge by coulomb counting method, for a fixed temperature and C-rate (a 2C-rate discharge takes half an hour). Table 2 presents the parameters used for the capacity tests in the investigated applications. This table shows the discharge regime used, the number of times this discharge is repeated and how the previous charge was achieved.

From Table 2 it can be noted that the discharging rates and temperatures used to define the battery capacity differ between RPT. The discharging rate used is usually closely related to the cell technology, and therefore to its nominal rate. Therefore, Sarasketa-Zabala *et al.* and Spagnol *et al.* use a 1C discharge rate to characterize their LFP cells [8], [12]. This chemistry is indeed characterized by its ability to deliver high powers. Braco *et al.*, Li *et al.* and Martinez-Laserna *et al.* characterized NMC batteries [7], [16], [17]. These batteries are generally used to fulfill high energy needs, and therefore their nominal current is lower than for LFP

batteries. This justifies the use of a lower discharge rate at C/3. For the charging process prior to the discharge, the purpose of this charge is to fully charge the battery. The charging rate is usually chosen as the nominal charge rate of the cell [8]. The resting time is rarely specified in RPT but is essential to ensure that the cell's relaxation processes are almost completed.

It is also interesting to note that there is no consensus on the characterization temperature, although most of these tests are performed at 25°C. This nominal temperature is generally different from the one used for ageing tests, 45°C for Han *et al.* [11] and 40°C for Li *et al.* [16].

Several protocols perform several identical discharges in a loop to obtain a more accurate capacity value. Sarasketa-Zabala *et al.*, Martinez-Laserna *et al.* and Belt *et al.* perform this test 3 times [8], [10], [17], while Han *et al.* even perform it 4 times [11]. However, while most authors consider only the last value obtained, Sarasketa-Zabala *et al.* and Han *et al.* average the values obtained to define the capacity of the cell [8], [11].

It can be noted that several protocols aim at defining capacities at several discharge rates. This helps to

TABLE 2: OVERVIEW OF CAPACITY TESTS

Reference	Charge	Cut-off	Discharge	Itera-	Rest
	rate	current	rate	tions	time
Braco <i>et al.</i> [7]	C/3	C/33	C/3	2	1h
Sarasketa- Zabala <i>et al.</i> [8]	1C	C/20	1C	3	-
Grolleau <i>et al.</i> [9]	-	C/20	C/10	1	-
Belt <i>et al.</i> [10]	-	-	1C	3	-
Han <i>et al.</i> [11]	C/3	-	C/3	4	1h
Spagnol <i>et al.</i> [12]	-	-	1C	1	-
Wang <i>et al.</i> [13]	-	-	C/20, C/2, 6C	2	-
Bloom <i>et al.</i> [14]	-	-	1C	1	-
Gering <i>et al.</i> [15]	C/10, C/5, C/2, 1C, 2C	-	C/10, C/5, C/2, 1C, 2C	1	-
Li et al. [16]	C/3	-	C/3	1	3h
Martinez- Laserna <i>et al.</i> [17]	C/3	-	C/3	3	-
Baure et Dubarry [18]	C/35, C/5, C/3	-	C/35, C/5, C/3	1	-

quantify the available amount of charge depending on the use of the cell. Grolleau *et al.* [9] chose to use a low discharge rate to estimate at the same time the OCV-SOC characteristic of the cell (see section IV).

#### III. RESISTANCE MEASUREMENT

The resistance measurement is often determined by the so-called pulse power test (PPT), hybrid pulse power characterization test (HPPC) or direct current internal resistance test (DCIR). They used square-wave current load to induce voltage drops allowing to measure the pulse resistance by ohm law. They are usually performed at different state-of-charge (SOC) levels and temperatures. Other methods like electrochemical impedance spectroscopy test (EIS) give access to the complex resistance of the cell. As it is rarely employed, this section will focus on PPT.

Like capacity test, PPT is also performed on most RPT protocols and allows to track the cell power fading. To define such a test, it is necessary to specify the SOClevels considered, the pulse intensities, the rest times imposed between pulses and after reaching a SOC-level. Table 3 presents all these information as well as the method used to switch from one SOC to another. In all the protocols introduced in this paper, the resistance characterization test is performed in discharge.

From Table 3, it can be noted that while the HPPC protocol is the most widely used, the setup used varies in all RPT. Indeed, some identify resistance values every 10% of SOC, while others prefer to estimate them every 15%, 20% or even only at specific levels. Thus, a compromise must be made between the desired accuracy and the time required to perform the test. For instance, to optimize this test time, Martinez-Laserna et al. [17] chose to determine the internal resistance value only at three main SOC-levels: the average SOC of their ageing cycle (50%) and their extreme SOC (80 and 20%). Furthermore, some authors have questioned the impact of this test on the cell ageing. Indeed, even if the duration is short, the intensity of the pulses can be high and have a significant impact. Gering et al. [15] preferred to perform a low-HPPC (L-HPPC) to determine resistance values at lower intensities than the proposed standards.

To precisely achieve the desired level of SOC, several methods are used. Wang *et al.* [13] consider the time and the discharge rate to obtain a specific change in SOC. Other RPT rather discharge the cell in CC-CV mode to a specific voltage level, equivalent to the targeted SOC and obtained by the OCV versus SOC characteristics. To increase the accuracy of the test, Sarasketa-Zabala *et al.* and Li *et al.* [8], [16] also added a SOC compensation phase between pulses. This compensation is performed at low current. Bloom *et al.* [14] considered the loss of SOC due to pulses in the discharge at the following SOC-levels.

It can also be noted that among all the selected authors, only Baure et Dubarry [18] did not perform a specific test to characterize the resistance of the cell. The latter preferred to estimate the value of the internal resistance from the capacity test. As they performed a very low current discharge (C/35) they were able to determine the OCV vs SOC characteristics of their cells. The value of the internal resistance was then estimated at different SOC-levels from the voltage drop between the C/35 and C/3 characteristics. If only the discharge resistance at C/3 was determined the time saving on this test is considerable.

Finally, Braco *et al.* [7] also considered only the discharge pulses. To simplify this test, their protocol consists of discharge phases to certain defined SOC-levels, followed by rest phases. The resistance values at SOC-levels are determined at the 10th second of each discharge phase.

TABLE 3: OVERVIEW OF PULSE POWER TESTS

Reference	SOC-	Pulses	Pulses	Rest	Rest
	levels	rates	time	time at	time
	(%)			SOC-	after
				level	pulse
Braco <i>et al</i> .	90→10	-	-	lh	-
[7]	$\Delta = 20$				
Sarasketa-	90→20	10C, 4C	17s	2h	5 min
Zabala et al.	$\Delta = 10$				
[8]					
Grolleau et al.	100→0	C/2 and ?	10s	45 min	45 min
[9]	$\Delta = 15$				
Belt et al. [10]	-	5C	-	-	-
Han et al. [11]	-	2C, 1C	30s / 10s	-	40s
			(Dch/Ch)		
Spagnol et al.	-	9C, 7C,	-	-	-
[12]		6C, 3C, 2C			
Wang et al.	100→0,	5C, 3.75C	18s-10s	1h	32
[13]	$\Delta = 10$				
Bloom et al.	90→10,	-	-	1h	-
[14]	$\Delta = 10$				
Gering et al.	-	-	-	-	-
[15]					
Li et al. [16]	80→20	-	10s	-	10 min
	$\Delta = 20$				
Martinez-	80-50-20	0.5C, 1C,	10s	-	-
Laserna et al.		1.5C, 2C,			
[17]		2.5C			

#### IV. OCV PROFILE DETERMINATION

Finally, the cell may also be characterized by its open circuit voltage. Most RPT therefore incorporate a test to determine this OCV or quasi-OCV in relation to the cell SOC. This characteristic allows with the help of electrochemical voltage spectroscopy (EVS) techniques to determine degradation modes of the cell. For quasi-OCV determination, two methods are mainly used. The low-rate test which is similar to the capacity test but with low current (>C/5), and the galvanostatic intermittent titration technique (GITT) which consists in discharging the battery to a specific SOC and waiting for the end of the relaxation phenomenon. Table 4 focuses on low-rate test mostly used here and shows the different current rates employed.

It can be observed that not all studies specifically performed OCV test as it can be time-consuming. However, both methods can be found. For instance, the quasi-OCV is performed by Bloom et al. [14]. They used a charge rate at C/25 while Sarasketa-Zabala et al. [8] performed it at a higher rate at C/5. The resistive drops are then more important and the accuracy of this characteristic is contestable. They justify this choice by indicating that it is the highest current rate that makes the plateaus to appear on their LFP cells. Li et al and Han et al. [11], [16] preferred to use the GITT method. Martinez-Laserna et al. [17] chose to determine the OCV both in charge and in discharge. This technique allows them to perform the cycle at higher rates without losing much accuracy on the characteristic. Indeed, the resistive losses during charging are supposed to compensate for

those during discharging by averaging the two obtained curves. In other RPT, the determination of OCV is obtained from capacity tests [9], [18] or PPT [9], [11].

#### V. RPT CONTENT AND SEQUENCING

While the capacity and pulse power tests are performed in all RPT, many other tests are implemented in the protocols selected. It is also relevant to note that the order of the main steps may differ. The protocols are thus defined in terms of their frequency and duration to avoid interrupting the ageing tests for a too long period and to avoid impacting too much the cell. In this perspective, some authors preferred to define two protocols: a detailed one (DC) performed occasionally, and a short one (SC) performed more frequently like every month of test. In this section, the different steps of the selected RPT are introduced. Table 5 summarizes the content and the sequencing of the different RPT in terms of DC and SC. Their execution order is given by the number which follow. As an example, "SC-2" in the capacity test column means that the corresponding reference use a short characterization which contains a capacity test played in position 2.

Among the tests performed in these protocols, all of them start either with a preconditioning phase: the cell performs a few complete cycles, or with the capacity test. However, as seen previously, this capacity test can be repeated several times. Some articles [8], [18] therefore include the preconditioning phase in the capacity test, thus repeated several times. It is also interesting to note that several RPT incorporate an EIS at different levels of SOC. Such a test is time consuming and requires specific equipment. Therefore, it is not systematically found. Han et al. [11] combined their HPPC test at different SOC-levels, with the OCV versus SOC test. According to the authors, this approach is very relevant to save time in RPT. However, it can easily lead to a bias in the obtained OCV values. Indeed, the compensation of the pulses must be perfectly realized, considering the impacts of the charge rates and the internal temperatures of the cell. Finally, other tests may be added in the periodic RPT. Spagnol et al. [12] performed the cold start test while Wang et al. [13] preferred to conduct the self-discharge test.

TABLE 4: OVERVIEW OF OCV-TESTS

Reference	Protocol	C-Rate	
Sarasketa-Zabala et al. [8]	quasi-OCV in discharge	C/5	
Bloom <i>et al.</i> [14]	quasi-OCV in discharge	C/25	
Gering et al. [15]	quasi-OCV in discharge	C/25	
Martinez-Laserna et al. [17]	quasi-OCV in charge and discharge	C/5	

Reference	Pre-	Capacity	Resistance	OCV vs	EIS	Cold start	Self-	Efficiency
	conditioning	test	test	SUC test			discharge	test
Braco et al. [7]		SC-1	SC-2	-	-	-	-	-
Sarasketa-Zabala et al. [8]	SC-1	SC-2	SC-4	SC-3	DC-5	-	-	-
	DC-1	DC-2/6	DC-4	DC-3				
Grolleau et al. [9]	-	SC-1	SC-2	SC-1	-	-	-	-
Belt et al. [10]	-	SC-1	SC-2	-	-	DC-4	DC-3	DC-5
		DC-1	DC-2					
Han <i>et al.</i> [11]	-	SC-1	SC-2	-	-	-	-	-
Spagnol et al. [12]	-	SC-1	SC-2/4	-	SC-5	SC-3	-	-
Wang et al. [13]	-	SC-1	SC-4		SC-3		SC-2	-
Bloom <i>et al.</i> [14]	-	SC-1	SC-2	DC-1	-	-	-	-
Gering et al. [15]	-	SC-1	SC-2	DC-1	SC-3		DC-2	-
Li et al. [16]	-	SC-1	DC-3	DC-2	-	-	-	-
		DC-1						
Martinez-Laserna et al. [17]	-	SC-1	SC-2	DC-3	DC-4	-	-	-
		DC-1	DC-2					
Baure et Dubarry [18]	SC-1	SC-2	SC-2	-	-	-	-	-

TABLE 5: OVERVIEW OF RPT CONTENT AND TEST SEQUENCES

It can also be noted that there are no standards on the periodicity of these tests. Some researchers prefer a frequent follow-up, like Li *et al.* [16] who performed it every week. Others [11], [17], [18], rather consider four weeks. However, it is relevant to observe that the frequency of testing is related to the aging test. Grolleau *et al.* [9] adapted the frequency according to the solicitation. They chose to perform the RPT on cells subjected to cycling tests twice as frequently as on cells subjected to be perform RPT more frequently on cells subjected to high current tests than on those subjected to low current regimes.

Detailed tests are quite relevant for tracking cell characteristics that do not require periodic observation or with low variation among time or cycles. Of the authors reviewed, six chose to add occasional detailed tests to characterize more accurately the cell. These tests helped define the characteristics of the cells at temperatures different from the nominal ones, classically around 25°C [8]–[10], [15]. It can be noted that most of them chose only three temperatures: a low one (5 or 10°C), a nominal one (around 25°C) and a high one (40 or 45°C). In the detailed RPT protocols, there are usually the time-consuming quasi-OCV [14]–[17], more comprehensive capability tests, or tests requiring specific hardware, such as the EIS [8], [17].

#### VI. RPT SYSTEMIC ANALYSIS

The RPT used in the selected aging studies are very different from each other. While some research focuses in comparing tests with one another, it is also relevant to compare the protocols as a whole. Indeed, the RPT must be optimized to obtain accurate information on the cells, while interrupting the aging tests as little as possible. To define them, compromises were made by the authors to achieve sufficiently accurate characterizations in a minimum of time. It is also essential to specify that the desired level of accuracy is closely related to the application considered. For instance, the SOC estimator proposed by Li *et al.* [16] requires regular and accurate monitoring of the evolution of the capacity. The precision on PPT is not essential, and this test is legitimately simplified. However, it is interesting to compare the different RPT in terms of the time they require and in terms of relevance of the figures obtained. This relevance is however very subjective to define. To quantify it, it was decided to assign a score to these protocols to compare them.

This score evaluates how the three main characteristics of the cell are obtained: its capacity, its internal resistance and its OCV curve. Each of these tests is scored out of 1. Points are also added to this score if additional tests have been carried out, for example EIS, cold cranking test etc. For the capacity test, the score obtained considers the number of times the test is repeated, the rate at which the discharge is performed and the way the cell is fully charged. For the power test, the score considers the number of SOC studied and the rest time at each level, the number of pulses and the rest time between each pulse. Finally, the OCV score is evaluated regarding the charge rate used. If the GITT method is used, the score considers the number of SOC assessed and the relaxation times imposed at each level. Therefore, these scores are an image of the RPT complexity but also relate to the amount of data collected. However, they are empirical but allow a comparison of RPT with each other based on quantitative criteria. Figure 1 shows the RPT classified according to their duration and the score they obtained.



Figure 1: RPT scores as a function of procedure time

Rationally, it can be observed that the highest-scoring RPT require the most time. It is interesting to note that the RPT used by Baure et Dubarry [18] does not present an interesting score/time trade-off compared to the other RPT and the criteria selected. Indeed, the latter only perform three capacity cycles, including a charge-discharge cycle at C/35. They do not assess the value of the internal resistance with a dedicated test.

However most periodic tests last between 10 and 20 hours. Thus, three RPT may be distinguished in terms of the time they consume. However, these protocols are repeated at a lower frequency than the others. It is therefore interesting to represent these same scores by considering both the duration they require, but also the frequency with which they are performed. It was therefore calculated for these protocols the proportion of the ageing test time spent for this RPT. This time, the score was re-evaluated by considering the RPT occasionally performed (usually at the beginning and end of the tests). Results are shown in Figure 2.

It can be observed that some RPT account for more than 10% of the ageing tests. This is considerable, as the impact of RPT on cell ageing may not be neglected [19]. Although the RPT of Braco *et al.* [7] is relatively short, it is repeated so frequently that it represents the RPT with the worst ratio of RPT time to aging test time. Wang *et al.*, Baure et Dubarry and Gering *et al.* [13], [15], [18] have very time-consuming RPT they repeat every month. Sarasketa-Zabala *et al.* and Spagnol *et al.* [8], [12] could not be included in Figure 2 because they do not specify the frequency with which they perform these RPT.

Regarding Figure 1 and Figure 2, four protocols can be distinguished. Gering *et al.* and Wang *et al.* [13], [15] have the highest scores. Belt *et al.* [10] presents the best compromise score / proportion of time dedicated to RPT, while Han *et al.* [11] have the best ratio score / time spent on RPT. It is therefore relevant to compare these four RPT according to the following criteria: periodic RPT score, global RPT score (including detailed RPT), periodic RPT time, global proportion of time dedicated to RPT, material needed and impact of the RPT on cell



Figure 2: Re-evaluated scores as a function of the ratio of the aging test time spent on cell characterization.

ageing. The Figure 3 compares the last four RPT according to these indicators. In this figure, the indicators have been normalized to the maximum achieved by the RPT.

Logically, Belt *et al.* and Wang *et al.* [10], [13] reach the maximum for score and proportion of time. However, they require more equipment than the other protocols to perform the EIS and low temperature tests. It can also be noted that they have a greater impact on cells than the other two RPT. Indeed, the -30°C tests and high rates used cause more degradations to the cells than the frequent but low-rate tests performed by Han *et al.* [11] or L-HPPC performed by Belt *et al.* [10] so as not to damage the cells. Finally, the reason that the RPT achieved by Han *et al.* [11] represents a high ratio of time test is that the discharge regimes used are quite low. The impact on ageing is therefore minimized compared to the ageing test performed.

#### VII. CONCLUSION

A case study review was performed in this paper based on twelve aging test campaigns. They were the support for comparison and discussion about the differences encountered in the definition of RPT procedures in the literature. Among the different test possibilities, the definition of an accurate RPT appears to



Figure 3: Strengths and weaknesses of four specific RPT

be a compromise mainly limited by hardware and time limitation. However, depending on the application and cells under consideration, these drawbacks may be lowered with the help of a smart testing plan. For example, splitting the procedure into short and detailed ones may reduce its impacts both in terms of time and undesirable aging.

Consequently, a unique RPT cannot be applied to all the studies. But the comparison of the different test parameters has put forward some gray areas. For example, the choice of the steps order or the resting time between steps are rarely explained. Deeper investigations are needed to evaluate these parameters in detail and create a testing common frame depending on the application or the cells considered.

To finish, as seen in the different tables presented above, RPT procedures used in literature are poorly documented. Many parameters are missing, equipment used are rarely mentioned and calculation methods barely explained. This makes it difficult to compare results between studies.

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