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STANDARD DEVELOPMENT WORK IN ISO TECHNICAL COMMITTEE 226 "MATERIALS FOR THE PRODUCTION OF PRIMARY ALUMINIUM"

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Abstract

Standard work aims to ensure that reliable analysis methods are available. ISO standards are commercially important and are recognised and used by suppliers and customers around the world. Reliable analysis enables the suppliers to describe their materials using common standards for well-established material properties, and the standards are a support for smelters when evaluating and comparing sources of materials.
ISO Technical Committee 226 maintains 110 standards on sampling and analysis covering smelter grade alumina, smelter grade fluorides, pitch, petroleum coke and solid carbon bodies including anodes and cathodes. The main work is done through dedicated work groups, one for each of the material groups. The need for modernisation of the standards is continuous as instruments improve and raw materials change. The paper will describe the committee, the work groups, current projects and ISO work in general. The paper is co-authored by Convenors and technical experts from the work groups. ***Aims of this paper are; to interest potential technical experts to participate in committee projects, and to encourage metal producing companies to support the important work that their participating staff makes within the committee.***

History, Administration

The current responsible ISO Technical Committee, ISO/TC 226, maintains 110 standards on sampling and analysis covering smelter grade alumina, smelter grade fluorides, coal tar pitch, petroleum coke and solid carbonaceous materials including carbon anodes and cathodes.
The standard development has mostly been done by the industry itself, with important contributions from academia, analysis and instrument developers. The standards represent a long time investment in technical expertise, and continued maintenance of this expertise, also inside companies' laboratories, is best done by taking part in the standard development and keeping up with current know how.
Work in this committee spans more than 40 years, counted from the first plenary meeting in Budapest in 1973; a full list of plenary meetings is given in Table 1.
Recent committee chairs have been Werner Schmidt-Hatting, Switzerland, to 2003, and then Harald A. Øye, NTNU and Norway, to 2011. The current Committee Chair is Lorentz Petter Lossius, Hydro Aluminium, Norway. Stéphane Sauvage is the Technical Programme Manager for TC226 on behalf of the ISO Central Secretariat, Geneva, and Knut Aune of Standards Norway is the Secretary to TC226 and the contact person for enquiries regarding the work (knut.aune@standard.no).

Table 1 Plenary meetings of the Technical Committee for materials for the production of primary aluminium, since 1973.

Venue	Date		
Wiesbaden (27)	2014-05-08	Montana-Vermala (13)	1993-05-11/13
Sierre (26)	2012-10-02/03	Rotterdam (12)	1991-05-14/16
Bratislava (25)	2011-04-06/07	Montréal (11)	1989-02-21/23
Aalesund (24)	2009-09-25/26	Milano (10)	1986-11-04/06
Beijing (23)	2008-04-25/27	Paris (9)	1984-10-23/25
Paris (22)	2006-09-26/28	Stockholm (8)	1983-06-22/30
Jeju, Korea (21)	2005-04-25/27	Zermatt (7)	1981-09-29/10-02
Tromsø (20)	2003-09-01/03	Oslo (6)	1980-05-20/23
Switzerland, Rigi (19)	2002-06-04/06	Köln (5)	1978-09-12/14
Delft (18)	2000-10-10/12	Capo Boi (4)	1977-05-10/13
Berlin (17)	1999-04-26/28	Paris (3)	1975-10-22/24
Seattle (16)	1997-09-15/18	London (2)	1974-05-20/23
Balestrand (15)	1996-06-10/14	Budapest (1)	1973-04-24/26
London (14)	1995-05-09/12		

The number of technical experts is not very large, and this paper, and a 2013 article in International Aluminium Journal[1] are both initiatives meant to attract the attention of potential new experts.

The committee has other documents than standards, and all such documents are published tagged with a sequential N-number, examples are results of voting, the resolutions from plenary meetings[2][3] and various informative documents[4].

Standard's Availability

The basis for ISO is sale of standards, both through the national standards authority and from ISO central. The standards in the TC226 program were until 2006 available only for single standard purchase. In 2006 a CD collection was published, made specifically for "Materials for the production of primary aluminium", at more reasonable cost per standard, filling a long time need. However, the CD gradually became out-of-date. Fortunately an even better solution is now ready; Stéphane Sauvage has informed the Committee that from 2014 an electronic collection is available from the ISO Store.
http://www.iso.org/iso/home/store/publication_item.htm?pid=PUB200007
As an annual subscription, this modern, electronic version has the advantage of being updated in parallel with the Committee work.

Committee and Work Groups

The most recent plenary meeting was hosted by German standardization authority, DIN, and SGL Carbon SE in Wiesbaden, Germany, May 8-9th, 2014. Participation was normal with eleven delegates representing seven of the twelve P-member countries attending. Time between plenary meetings is 1.5 to 2 years, and in the interval, standard development goes on in Work Groups. This work is organised with a dedicated Work Group (WG) responsible for each material group listed above. The Work Group is organised with a Convenor and Technical Experts, and after the Wiesbaden meeting the convenorships were unchanged. The current Work Group Convenors are:

- Ray Brown of Alcoa World Alumina, Australia; WG3 on Smelter Grade Alumina
- Lin Wu and Xujin Xue of Do-Fluoride Chemicals Co., Ltd., China; WG4 on Smelter Grade Fluorides
- Nigel Turner of Koppers UK Ltd., United Kingdom, WG1 on Coal Tar Pitch
- Professor Harald A. Øye of NTNU, Norway; WG2 on Solid Carbonaceous Materials and Ramming Paste
- Jean-Claude Fischer, R&D Carbon Ltd., Switzerland; WG6 on Petroleum Coke

The main task of the Committee is to maintain and update the existing standards. All 110 standards are updated according to a 5-year cycle of Systematic Reviews, giving, on average, 22 standards to review each year, 4-5 standards per Work Group. This is routine work and progresses well.

Figure 1 Overview of standard development process. /4/

The Secretariat and Committee Chair is the link between ISO and the Work Group and the Technical Experts. The actual writing of the technical part is done by a Convenor or Work Group expert, who then sends text and figures to the Secretariat for ISO formatting. ***Note that Technical Experts do not need experience with ISO formatting or ISO formalities.*** The experts can therefore spend time on the analysis methods rather than use the time on formatting. The ISO series of ballots ensure that all interested parties can review and suggest changes to the drafts.

Another important task for the Committee is the development of new standards, and this work often interests the technical experts the most. New standards evolve due to improved experience and new or improved analysis instrumentation. Sometimes a new standard can replaces out-of-date methods with new and improved methods with the aim to later withdraw the out-of-date methods. Standards that are withdrawn are still available. Such is the case for the long-awaited new XRF elemental analysis standard ISO 23201 is through Draft stage and awaits decision for final ballot or direct to publication. It will replace ten wet chemical analysis standards.

ISO Voting

Using the Systematic Review as an example, for the technical expert the involvement consists of getting notification four times annually from the national ISO standardization authority of pending reviews. Voting in ISO is by P-Member country, one vote per country, organised by the national standardization authority. After being notified of a pending vote, the Technical Expert gives a recommendation on how the national ISO standardization authority should vote. For TC226, the number of Technical Experts per country and material work group is low and the voting is mostly unanimous.
Note that the 5-year review is often used as an opportunity to initiate a revision, but a revision can be initiated by the Technical Expert and national standardization authority at any time. For an ISO Technical Expert, the work in the committee and work group gives many opportunities to improve and correct the standards, and also to develop new standards.

Smelter Grade Alumina, Convenor Ray Brown

The Smelter Grade Alumina (SGA) committee, ISO/TC226/WG3 has been active for over 15 years. The current Convenor is Raymond Brown. Mr. Brown has held the chairman position for the Australian Standards SGA committee MN9 for eight years; he is a Senior Chemist based in Australia.

Physical and chemical properties of alumina are characterised by alumina suppliers to enable customers to assess the suitability and potential impact an alumina will have on their smelting process. Most of the modern methods are based on methods developed by the Australian Standards alumina committee, MN9. These methods have involved international participation in their development. Over the last eight years new/updated methods have been published for measuring particle sizing by electroform sieves, ISO 2926, determination of attrition index (a property that describes an alumina's propensity to breakdown on transportation), ISO 17500, and calculated Al_2O_3 content in SGA, ISO 12315. See the table for a full list.

In the next year a number of new methods will be published, these include methods for bulk density (tapped and untapped), flowtime (a measure of alumina's flow characteristics), alpha-alumina content (by XRD), and the Work Groups most challenging project, elemental analysis by XRF spectrometry.

Table 2 Methods in the AS 2879 Series developed by the Australian Standards alumina committee, MN9, and the corresponding ISO method as of September 2014.

Part 1: Determination of loss of mass at 300°C and 1000°C	ISO 806:2004 (Ed. 2) Aluminium oxide primarily used for the production of aluminium — Determination of loss of mass at 300 °C and 1 000 °C
Part 2: Determination of particles passing a 20 μm aperture sieve	ISO 23202:2006 Aluminium oxide used for the production of aluminium - Determination of particles passing a 20 micrometre aperture sieve
Part 3: Determination of alpha alumina content by X-ray diffraction	ISO/NP 19950 Aluminium oxide primarily used for the production of aluminium — Determination of alpha alumina content — Method using X-ray diffraction net peak areas
Part 4: Determination of specific surface area by nitrogen adsorption	ISO 8008:2005 (Ed. 2) Aluminium oxide primarily used for the production of aluminium -- Determination of specific surface area by nitrogen adsorption
Part 5: Alumina - Determination of Angle of Flow	No ISO method
Part 6: Determination of the mass distribution of particle sizes using electroformed sieves	ISO 2926 Aluminium oxide used for the production of primary aluminium — Particle size analysis for the range 45 μm to 150 μm — Method using electroformed sieves
Part 7: Determination of trace elements— Wavelength dispersive X-ray fluorescence spectrometric Method	ISO/WD 23201.2 Aluminium oxide primarily used for the production of aluminium — Determination of trace elements — Wavelength dispersive X-ray fluorescence spectrometric method
Part 8: Determination of bulk density	ISO/DIS 18842 Aluminium oxide primarily used for the production of aluminium — Method for the determination of tapped and untapped density
Part 9: Determination of flow time	ISO/DIS 18843 Aluminium oxide primarily used for the production of aluminium -- Method for the determination of flow time
Part 10: Determination of attrition index	ISO 17500:2006 Aluminium oxide used for the production of primary aluminium — Determination of attrition index
Part 11: Guide to reporting Al2O3 content of smelter-grade alumina	ISO 12315:2010 (Ed. 1) Aluminium oxide primarily used for production of aluminium -- Method for calculating the Al2O3 content of smelter-grade alumina
Part 12: Determination of the angle of Repose	ISO 902: 1976 Aluminium oxide primarily used for the production of aluminium - Measurement of the angle of repose

The XRF method covers the analysis of fourteen elements at trace and minor concentration levels, lithium borate fusions are used to prepare specimens for XRF analysis. Calibrations are made using a first principles calibration methodology (based on weighed amounts of high purity chemicals). Validation of analyses is done using two reference materials, one of which was developed by the Australian Standards alumina committee, MN9 specifically for this purpose (known as ASCRM027). It is a comprehensive method with a proven capability. It is based on an Australian Standard method that has been modernised, extended and improved. Publication of this method is expected in 2015, it will formally make obsolete the many wet chemical analytical ISO methods used for determination of trace elements in SGA.

With the publishing of these methods the global industry will have a set of modern, capable, well supported reference methods that cover all the most important properties of alumina that the industry uses to comprehensibly characterise alumina. These methods produce data that is accurate and precise to within known, and acceptable tolerances, this data can be compared between laboratories and markets.

Planned work includes the development of a method for preparation of alumina laboratory samples.

Smelter Grade Fluorides, Convenor Lin Wu

Work Group 4 on Smelter Grade Fluorides today faces considerable challenges. Most of the existing ISO standards relevant to Smelter Grade Fluoride analysis are old and were published around 1970-1980, and the chemical property determinations are based on wet chemical methods not practical today.

The present Convenor of WG4 in ISO/TC 226 is Mr. Lin Wu (SAC), and the committee Technical Experts are L.P. Lossius (SN), Ray Brown (SA), Josef Lovcican (SUTN) and Xujin Xue (SAC). Mr. Lin Wu and Mr. Xujin Xue are both from Do-Fluoride Chemicals Co., Ltd., a professional supplier of smelter grade fluoride in China, with fluoride products such as Anhydrous Aluminium Fluoride and Cryolite for global customers. Do-Fluoride Chemicals also supplies advanced technical solutions and services including sets of certified reference materials for Aluminium Fluoride, Cryolite, Aluminium Electrolyte Bath, Magnesium Fluoride and Sodium Fluoride. In China, Do-Fluoride Chemicals has lead or participated in more than 50 projects involving Chinese industrial and national standards from individual analytical method to product classification. This background is useful in the work for new Smelter Grade Fluoride standards.

The Convenor and Work Group are addressing the problem of old standards, beginning with replacing old wet chemical element analysis with modern X-Ray Fluorescence spectrometer (XRF) methods. The major issues are time and precision; the wet methods take too much time and manpower for a complete element analysis. For example, a full wet chemical analysis of all the trace elements in an aluminium fluoride sample (F, Al, Na, Si, S, P, Fe) would usually take at least 4 laboratory operators working for 8 hours. And the precision and stability of the results can be easily affected due to the complicated procedures and the manual factors. To compare, using the trace element analysis in aluminium fluoride as an example again, an XRF can determine all the relevant elements (F, Al, Na, Si, S, P, Fe) within only 15 minutes after receiving the sample - without consuming the test portion.

The first part of establishing XRF based elemental analysis was completed in 2012 with a new standard for major and trace element analysis of aluminium fluoride, AlF_3.

- ISO12926:2012 Aluminium fluoride for industrial use — Determination of trace elements — Wavelength dispersive X-ray fluorescence spectrometric method using pressed powder tablets.

Besides the analysis of chemical properties, there are methods relevant to physical properties as well that are important for the performance in aluminium smelters. This part is somehow a vacancy in the ISO system and new standards are in priority to be established. WG4 is continuing working on new work items including

1. Cryolite elemental analysis by XRF
2. Sampling and storage of AlF_3 and Cryolite
3. AlF_3 purity
4. AlF_3 loss on ignition
5. AlF_3 bulk density

Summing up, WG4 – the Work Group of smelter grade fluorides - is dedicated to review existing ISO standards, in order to find the necessary new standards according to the development of modern analytical technologies relevant to fluorides. By participating in ISO/TC226 projects since 2005, Mr. Lin Wu and Mr. Xujin Xue continuously support the ISO standard development of Smelter Grade Fluorides and the work group for fluorides is looking forward to more support from global colleagues.

Standard Development, Cathodes

ISO/TC226 Work Group 2 for Solid Carbonaceous Materials had a very active period 1990 – 2007. A detailed description of the activities was given by then TC226 Chairperson Harald A Øye in a 2008 TMS presentation/5/. Professor Harald A. Øye is today Convenor of WG2 and professor emeritus at the Norwegian University of Technology, NTNU.
In the 1990'ties it was found that for cathode materials (blocks and ramming paste) it was important to characterize properties at operational conditions. Examples of standards developed for this are sodium expansion for cathode blocks with and without pressure, and expansion/shrinkage of ramming paste during baking.

- ISO 14428 Ramming paste expansion/shrinkage during baking
- ISO 15379-1 Cathode expansion due to sodium penetration with application of pressure
- ISO 15379-2 Cathode expansion due to sodium penetration without application of pressure.

For thermal conductivity, electrical resistance and strength only properties at ambient temperature are ISO standardized. The temperature coefficient as well as change with time are however very different for the different materials. Details for use in modelling work from this development can be found in the book "Cathodes in Aluminium Electrolysis"/6/. This book is also published in Russian and Chinese languages. From 2007 mostly routine 5-year systematic reviews were performed in WG2, but since 2011 the group has become more active. Current activities are harmonising mechanical strength standards (see below) and supporting development of a standard on abrasion of carbon cathodes.
For abrasions resistance, the current standard development is focused on comparing several methods for carbon cathode abrasion, to establish an agreement on a common method suitable for ISO standardization. The tests have been run on amorphous, graphitic grades and graphitized cathodes, as well as on needle coke graphitized electrodes to determine both if the abrasion is a useful measure for the prediction of the lifetime of cathodes and to discuss different laboratories' approach. Participating laboratories and developers include R&D Carbon, SGL Carbon and the Materials Science department at NTNU in Norway. The RDC 191 equipment (dry abrasion) has been compared to two methods in an alumina slurry. The results are being evaluated.

Anodes and Cathodes, Technical Expert Andreas Schnittker

The SGL Group has been involved in various national and international technical committees for the establishment and further development of industry standards for many years. The benefits for the SGL Group include mandatory uniform test standards for our raw materials specifications and for data sheets and specifications for our customers. It is almost impossible to imagine the global business of the SGL Group without international standards. The core reasoning, as described by Andreas Schnittker, Quality Leader of the SGL Group: "*We would have to discuss each test procedure with every supplier and customer on an individual basis. What's more, we would have lots of discussions with our business partners regarding non comparable test results. By actively helping to design industry standards, we also take on partial responsibility for the efficient further development of the sector. If you don't standardize, you'll be standardized!*" For SGL Carbon, test standards are used at the central laboratory in Meitingen as well as at the CFL, GCE, and GS business units.

Andreas Schnittker is the chairperson of the DIN standardization committee for "Carbon materials" and was host to this year's Plenary when representatives of the "ISO Technical Committee 226" work groups met at the SGL Group location in Wiesbaden (Germany).
Examples of ISO standards for the testing of materials for the aluminum industry that are critical to the business are

- ISO 8005 Coke ash content
- ISO 6998 Pitch coking value
- ISO 12984 Coke particle size distribution
- ISO 12985 Apparent density
- ISO 14420 Coefficient of thermal expansion (CTE)

The revision of certain test standards was agreed upon at the meeting, including the updating and harmonizing of strength standards under the leadership of the SGL Group:

- ISO 12986-1 Three-point method for bending/shear strength
- ISO 12986-2 Four-point method for flexural strength
- ISO 18515 Compressive strength

Petroleum Coke, Convenor Jean-Claude Fischer

The Petroleum Coke Work Group, WG6, was established at the 2012 Sierre plenary meeting of TC226 to decrease the number of standards in WG2 (solid carbonaceous materials) from a very high 50 standards to a more manageable 30 standards. Some standards cover both petroleum coke, anodes and cathodes, but this overlap has as yet not been problematic.
The Convenor of WG6, Petroleum Coke, is Jean-Claude Fischer of R&D Carbon Ltd, Sierre, Switzerland. R&D Carbon has been active in the ISO committee since 1986, and even before that, from 1966 onwards, the R&D Carbon management was involved in standardization of DIN methods for DKG, Deutsche Keramische Gesellschaft. Several of the DIN methods developed for petroleum coke, pitch, anodes, cathodes and graphite were subsequently adopted as ISO standards. In addition to standard development, R&D Carbon is known as a supplier of equipment and calibration standards to execute analysis according to the DIN/ISO methods. Worldwide, 1700 such testing instruments are used in the carbon business.

The following is a list of ISO Petroleum Coke Standards in Revision or development as of September 2014.

Calcined petroleum coke always must undergo a de-oiling step to define if and how much de-dusting agent has been added. The methods for the determination of the oil content ISO 8723 (dichloromethane washing) and ISO 6997 (heating method) are being analyzed for their efficiency with water soluble de-dusting agents.

- ISO 8723 Calcined coke – Determination of oil content – Method by solvent extraction
- ISO 6997 Calcined coke – Determination of apparent oil content – Heating method

New precision figures for ISO 10236 have been updated to the latest round robin results. Repeatability is now at 0.01 g/cm³ reproducibility at 0.03 g/cm³.

- ISO 10236 Green coke and calcined coke for electrodes – Determination of bulk density (tapped)

The recently published ISO 10143 is a revision of the 1995 version where the grain size used for petroleum coke have been adapted to the petroleum coke analysis methods.

- ISO 10143 Calcined coke for electrodes – Determination of the electrical resistivity of granules

Currently TC226 is monitoring the use of Normative References in standards, to determine if the reference is actually required for use of the standards and has to be acquired by the laboratory. In cases where there are too many normative references, as for ISO 12984, coke grain size distribution, the standard is revised.

- ISO 12984 Calcined coke – Determination of particle size distribution

The standards are used routinely at the R&D Carbon research center generating knowledge for the development of the necessary equipment. The company determines repeatability and reproducibility figures through international round robins.

Coal Tar Pitch, Convenor Nigel Turner

Working Group 1 (WG 1) has primary responsibility in ISO committee TC 226 for pitch as a raw material for anodes, cathodes and sidewalls in the aluminium smelting industry. There are fourteen current Standards for which WG 1 is custodian. Under the leadership of Convenor Nigel Turner of Koppers UK, the standards are all systematically reviewed in detail by WG 1 members for their technical and editorial content. In 2014 seven of the fourteen Standards were given this in-depth review with recommended revisions delivered to the ISO Secretariat for implementation.

Pitch is a very complex material in its composition and contains more than a thousand different molecular components. For this reason it is particularly important to have highly dependable, standardized methods of test that will give reliably comparable results within a laboratory and between laboratories.

There is close contact with ASTM subcommittee D02.05 with reference to general aims and objectives.

New tests and methods to answer today's and tomorrow's questions from the aluminium smelting industry about pitch properties and performance are particularly important for WG1, especially in the next one to three years.

There are plans for three new standards and work is in progress.

Measuring trace elements in pitch by X-ray fluorescence (XRF), inductively coupled plasma atomic emission spectroscopy (ICP-AES) and atomic absorption spectroscopy (AA) as a projected three-part, single standard; the single-standard approach is comprehensive and also avoids duplicating of some of the content.

Measuring the apparent density of pitch by a simple buoyancy (Archimedes) method; this will become a rapid and efficient alternative to the existing ISO standard for using a helium pycnometer because many laboratories do not have access to this equipment. Modern density balances are being included as an option within the standard. Density conversion to different temperatures according to requirements is also being included.

Measuring directly the volatility of pitch by a simple empirical method to enable the quantity of volatiles emitted from a particular pitch at a range of set temperatures. The focus is completely on measuring the percentage of volatile emitted because it is the most relevant metric for industrial usage and conditions, such as in a paste plant. Simplicity and ease of use are primary objectives in the development of this standard.

The ISO Committee in General

Considerable work is needed to maintain the standards, and as described, maintaining and developing is an international cooperation. The last two plenary meetings were hosted by the Swiss standardization authority SNV in October 2012, and the German standardization authority DIN in May 2014. Participation was both times good with more than half the P-member (voting members) countries attending. A full list of plenary meetings was given in Table 1 and the list of current P-members is given in Table 3.

Table 3 ISO P-Member Countries with abbreviated name of the ISO standardization authority

Australia (SA)
China (SAC)
Germany (DIN)
Great Britain (BSI)
Libya (LNCSM)
Netherlands (NEN)
Norway (SN)
Republic of Korea (KATS)
Russian Federation (GOST R)
Slovakia (SUTN)
Spain (AENOR)
Switzerland (SNV)

Final Notes on Advantages of ISO Work Participation

ISO work is a combination of idealistic effort and professional expertise and depends much on the good will of the companies supporting the work. There are many advantages to taking part.

- A significant return of value is the inter-laboratory studies. An important part of standardization work is to establish the obligatory precision statement. Taking part in ISO work for this reason leads to opportunities for participation in Inter-Laboratory Studies and Round Robins with other, highly qualified laboratories.
- Modern instruments are complex, so participation in method development is helpful for establishing good internal instrumental analysis standard practices in a company. This means getting to learn details of the analysis methods.
- There is always a formal and informal side to standard work; and also opportunities for learning of issues with methods from other users not generally available.
- Developing a company's own sampling and sample preparation can be costly; methods adapted for standardization for sampling represent the backbone of such methods.
- The new analysis methods considered for standardization might be new, cutting edge technology; users such as metal plants might not learn of such methods otherwise.
- Analysis using standards means establishing a reliable history of analysis with commercial partners; this will allow reliability and durability, stability and non-change over time.
- ISO standardization work is important for analysis quality and supports reliable supplier-customer relationships. The quantified analysis value is a cornerstone in communication.
- ISO is grateful to the companies that allow scientific personnel to take part in standard development and maintenance, including the laboratory resources necessary. We would appreciate more companies encouraging their analytical personnel to join this work.

Acknowledgement

ISO Technical Committee TC226 "Materials for the production of primary aluminium" is responsible for standards for smelter grade alumina, smelter grade fluorides, anodes, petroleum coke, pitch, cathodes and carbonaceous lining. ***It is hoped that the paper shows the importance and usefulness of having active ISO Technical Experts in your company.***

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