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Bromine-enhanced Mercury Abatement from Combustion Flue Gases – Recent Industrial Applications and Laboratory Research

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Bromine-enhanced Mercury Abatement from Combustion Flue Gases – Recent Industrial Applications and Laboratory Research*

Kurzfassung

Bromgestützte Quecksilberabscheidung aus Verbrennungsabgasen – Neuere industrielle Anwendungen und Laborforschung

Der Beitrag stellt die bromgestützte Quecksilberabscheidung vor. Die neuartige, kostengünstige Technologie fußt auf der Hg-Bromierung im Rauchgas auf seinem Weg durch Kessel und z.B. SCR-Katalysatorbett sowie Staubfilter. Bromhaltige Verbindungen wie wässrige Lösungen von Bromidsalzen werden dem Hauptbrennstoff (z. B. der Kohle) zugesetzt oder direkt in den Feuerraum injiziert, wo sie mit dem (z. B. kohlestämmigen) SO_2 reagieren (Sulfatation der Bromide) und somit HBr/Br_2 für die angestrebte Quecksilberoxidation freisetzen. Das in 2000 erfundene Verfahren wurde von Bayer Industry Services GmbH & Co. oHG (BIS) in 2002 zum Patent angemeldet; kürzlich sind das US-amerikanische Patent US 6,878,358 und das deutsche Patent DE 102331 erteilt worden. Dem EPRI/USA wurde eine F&E-Lizenz zur Durchführung von Demonstrationsversuchen in den USA erteilt. Inzwischen wurde die Technologie im Rahmen von DOE-geförderten Demonstrationsprojekten an kohlegefeuerten Großkesseln mit beachtlichem Erfolg getestet.

Introduction

The new process of bromine enhanced mercury abatement [1] was discovered by Vosteen and Pohontsch in 2000 [2] and studied in more detail by Kanefke and Nolte in

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2001/2002 [3, 4] and since then constantly applied industrially at the Hazardous Waste Combustors (HWC) of BIS, Figure 1. Since August 2004 the new process is constantly applied, too, at two Stationary Fluidised Bed Combustors (SFBC) for the mono-combustion of municipal sewage sludge of Emschergerossenschaft, Essen/Bottrop (Germany) [5]. The two SFCB plants in Bottrop, each equipped with heat recovery boiler, ESP and two-staged scrubber (acidic and neutral), primarily used the expensive NaClO_2 as oxidising agent injected at the scrubber entrance, but did now change over to aqueous sodium bromide injected into the hot SFBC free board. Changing over to this comparatively cheap and easy-to-handle oxidising agent has cut down the operating costs of both plants considerably.

In-plant Research

BAYER Industry Services GmbH & Co oHG operates at its German sites Leverkusen, Dormagen and Uerdingen four rotary kiln units for hazardous waste incineration. Two of them are located at the BAYER waste management centre in Leverkusen-Bürrig. The four units have a similar, BAYER-typical design as shown in Figure 1. Since 2000 extensive mercury-related in-plant research [2 to 4] has been carried out at the Leverkusen HWC kilns. Results on adsorption and oxida-

tion (elution) of mercury at the tail-end SCR-DENOX-catalyst bed have already been published in detail [6].

During almost all test runs the flue gas was spiked more or less with mercury, either continuously (up to $20,000 \mu\text{g}/\text{Nm}^3$, dry) or discontinuously (up to some $200,000 \mu\text{g}/\text{Nm}^3$, dry): Mercury in form of an aqueous HgCl_2 solution was injected continuously at the head of the rotary kiln or at the top of the post-combustion chamber; at the high combustion temperatures, all HgCl_2 added is directly converted into Hg_{met} . On the other hand, metallic mercury as such was injected “discontinuously” via the oven inspection port near to the bottom of the post-combustion chamber; this was achieved by throwing in small plastic bottles filled with mercury (“Hg bombs” of 5 g to 340 g Hg_{met} every 5 minutes), Figure 2.

Gaseous mercury was continuously measured by different CEMs (continuously measuring emission control monitors) installed at different places (details see [6], there Figure 2, showing four measuring points in front of and behind the tail-end SCR as well as in front of and behind the catalyst beds inside), e.g. a) in the scrubbed flue gas behind the Condensation Electrostatic Precipitator (CESP) and b) in front of the tail-end SCR-DENOX catalyst-bed, c) behind the tail-end SCR-DENOX catalyst-bed and d) in the clean gas behind the tail-end SCR-DENOX.

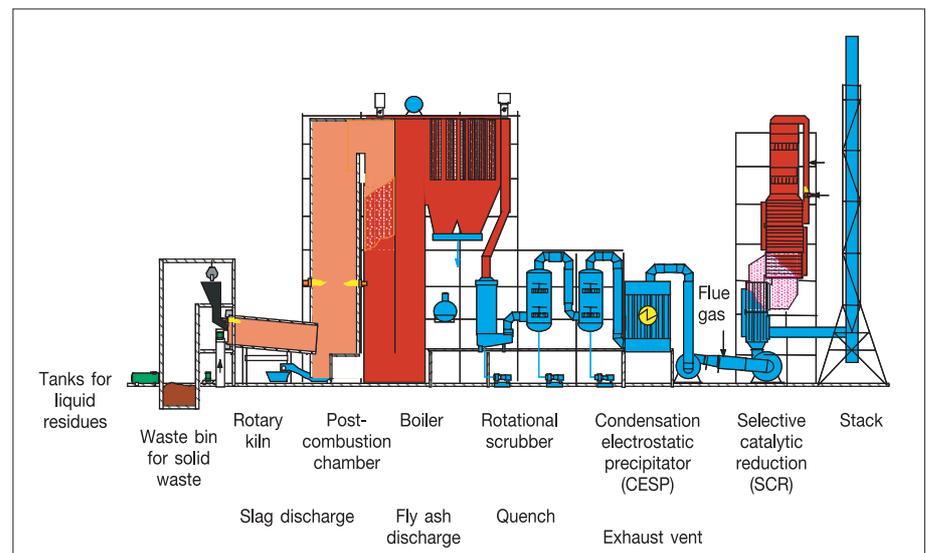


Figure 1. Hazardous Waste Combustor KILN A of BAYER Industry Services GmbH & Co. oHG in Leverkusen, Germany.

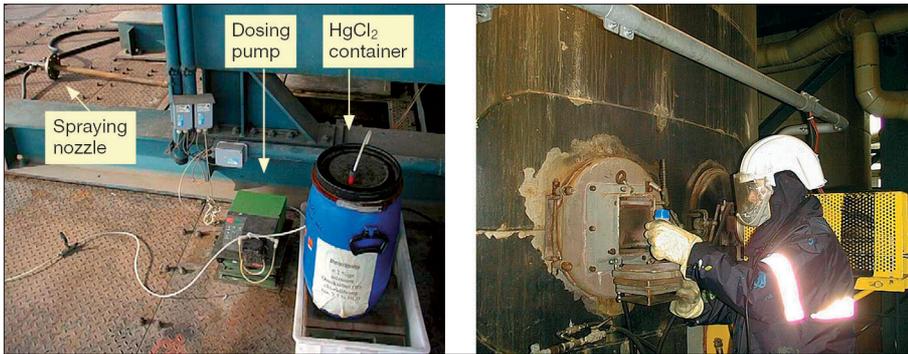


Figure 2. Continuous mercury injection on top of the post-combustion chamber (left hand) and discontinuous mercury injection (Hg-bombs) at its bottom (right hand).

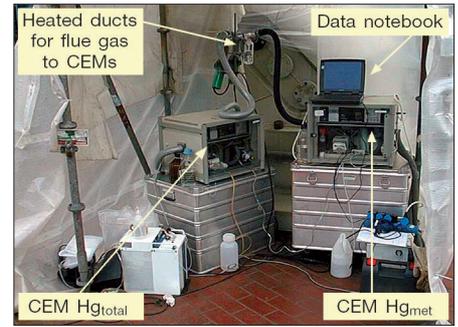


Figure 3. Continuous measuring of total mercury or elemental mercury (details see [6]).

At these measuring points from time to time CEMs were measuring simultaneously for mercury speciation, i.e. they were working in parallel either without reductive gas pre-treatment (no reduction of Hg^{2+} for measurement of Hg_{met} only) or with reductive gas pre-treatment (reduction of all Hg^{2+} to Hg_{met} for mea-

surement of Hg_{total}), followed by a Hg_{met} -monitor (based on cold vapour AAS), see Figure 3. The official Hg_{total} emission control at the stack was equipped with a SCEM (semi-continuous measuring emission control monitor) working with reductive gas pre-treatment by $SnCl_2$ and applying gold amalgamation.

As described in [1], bromine was added by co-combustion of bromine-rich (and chlorine-rich) wastes (normal HWC working conditions) or by injection of diluted HBr or aqueous NaBr (exceptional HWC working conditions). During co-combustion of highly halogenated wastes controlled sulphur-addition in the form of granulated sulphur was applied as described in [9] (see also [2], [7] and [8]).

Applying Br/Hg mass ratios > 300 complete mercury oxidation was achieved, i.e. all mercury in the boiler exit flue gas was converted in its water-soluble ionic form Hg^{2+} (100 % Hg_{ion} , 0 % Hg_{met}), mainly as $HgBr_2$ and some $HgCl_2$; therefore in the existing multistage scrubbing system excellent mercury removal (abatement efficiency $\eta > 99.8$ %) was realised, Figure 4.

Test runs at the HWC plants of BIS in Leverkusen-Bürrig and later at a PC-fired wet bottom boiler of BAYER AG in Uerdingen (as shown in Figure 13) have been demonstrating that bromine is much more effective in Hg-oxidation than chlorine - under high Cl_{total} -loads as in waste incineration at least > 25 times more and up to > 100 times more under low Cl_{total} -loads as in coal combustion [1], [10], Figure 5.

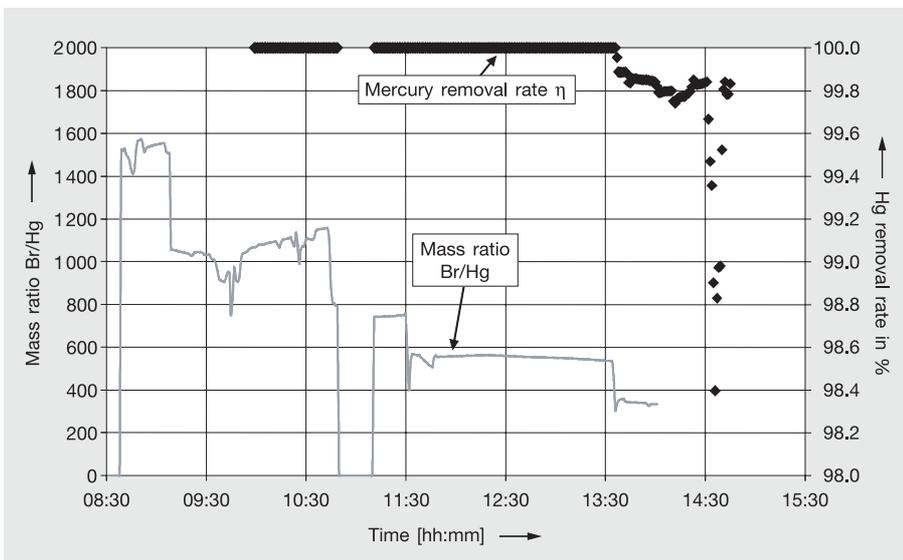


Figure 4. Influence of the mass ratio Br/Hg on mercury removal efficiency η of the wet multistage flue gas cleaning system of the HWC (9600 $\mu g/Nm^3$ dry raw gas mercury concentration by continuous injection of a HCl-stabilised $HgCl_2$ -solution).

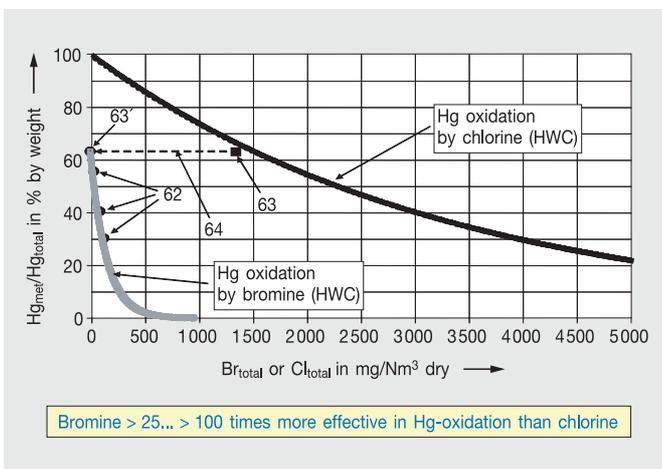


Figure 5. Mercury oxidation in the HWC boiler flue gas by chlorine (mainly existing as HCl) and by bromine (mainly existing as Br_2) [1].

Chlorine-enhanced Hg-oxidation

Relevant global reactions

$4 HCl + O_2 \leftrightarrow 2 H_2O + 2 Cl_2$	Chlorine-Deacon-reaction
$SO_2 + Cl_2 + H_2O \leftrightarrow SO_3 + 2 HCl$	Chlorine-Griffin-reaction
$SO_2 + \frac{1}{2} O_2 \leftrightarrow SO_3$	SO_2/SO_3 -conversion
$Hg + Cl_2 \leftrightarrow HgCl_2$	Direct Hg chlorination

Chlorine less effective in Hg-oxidation
 because $Cl_2/Cl_{total} \ll 1$
 (e.g. < 4% in HWC and < 1% in coal combustion)
 SO_2 is consuming Cl_2 during boiler passage

Figure 6. Relevant global reactions of mercury chlorination in boiler flue gases.

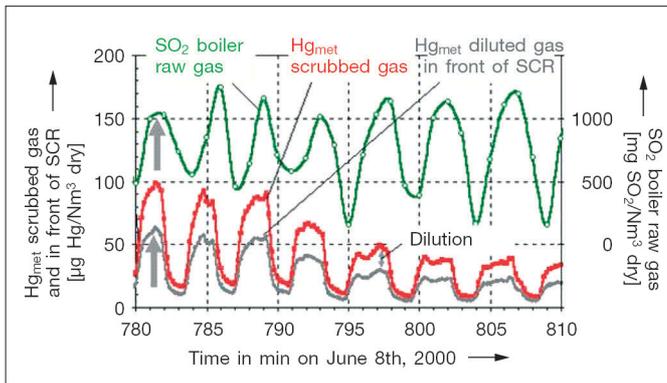


Figure 7. Artificially-induced oscillations of the SO_2 concentration in the boiler raw gas are inducing concurrent oscillations of the Hg_{met} -concentration in case of high chlorine loads (around $20,000 \text{ mg Cl}_{\text{total}}/\text{Nm}^3$ in boiler raw gas) [2], [10].

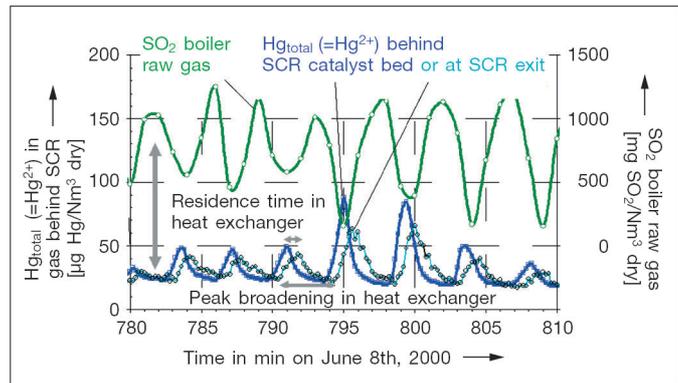


Figure 8. Artificially-induced oscillations of the SO_2 concentration in case of high chlorine loads in the boiler raw gas are inducing further down stream countercurrent oscillations of the $\text{Hg}_{\text{total}} (= \text{Hg}^{2+})$ -concentration in the cleaned gas behind SCR-DENOX catalyst bed [2], [10].

Thermodynamics and Kinetics

Chlorine-based mercury oxidation is governed by four simultaneous global reactions, Figure 6. For kinetical reasons only the direct mercury oxidation by Cl_2 has to be considered ($\text{Hg} + \text{Cl}_2 \rightarrow \text{HgCl}_2$), while the so-called indirect mercury oxidation ($\text{Hg} + 2 \text{HCl} + 0.5 \text{O}_2 \rightarrow \text{HgCl}_2 + \text{H}_2\text{O}$) is staying irrelevant. This important fact has been demonstrated by a fascinating “in-plant experiment”, Figure 7 and Figure 8. The boiler raw gas was containing about $20,000 \text{ mg HCl}/\text{Nm}^3$ dry, while the artificial oscillations of the SO_2 concentration were induced by corresponding oscillations of sulphur addition to the rotary kiln [2], [10]. Figure 7 is showing the resulting concurrent oscillations of the Hg_{met} -concentrations in the scrubbed flue gas behind the CESP as well as in front of the tail-end SCR-DENOX – after dilution by mixing with the scrubbed flue gases of the neighbouring HWC plant. In Figure 7 it becomes (indirectly) visible that SO_2 is consuming Cl_2 with the consequence that Cl_2 is lacking for mercury chlorination: Hg_{met} is going up or down when SO_2 is going up or down respectively.

Figure 8 is showing the resulting countercurrent oscillations of the Hg_{total} (mainly oxidised) concentration behind the SCR-DENOX catalyst bed as well as behind the heat exchanger at the SCR-DENOX exit. This - at first sight astonishing - behaviour is (indirectly) showing the fact that those four global reactions, governing already the mercury oxidation in the boiler raw gas (containing e.g. a certain fly ash metal oxide inventory as catalysts), are also governing the mercury oxidation in a SCR-DENOX catalyst bed, see remarks further down.

For modelling of mercury oxidation in waste incineration boilers as well as in power station boilers, it is necessary to combine equi-

librium thermodynamics (*Gibbs* free enthalpy minimisation) of those four homogeneous (uncatalysed) or heterogeneously catalysed global reactions with an adequate modelling of the corresponding chemical kinetics. This has been achieved for mercury chlorination following the so-called “stop temperature method of *Vosteen*”, which for in-plant experiments is giving way to realistic results [5]. Above its individual stop temperature, any reaction is running in its equilibrium, below its stop temperature the reaction is not going on anymore (“frozen”). Thus, the simplifying modelling (“only global reactions considered”) is still fairly realistic (“halogenes and SO_2 sufficiently considered”) and complex; not only linearly independent reactions as e.g. the chlorine *Deacon* reaction and the chlorine *Griffin* reaction are taken into account, but linearly dependent reactions, too (as the direct SO_2/SO_3 conversion).

Free chlorine is needed for direct mercury chlorination, Figure 6. At high temperatures the equilibrium of the chlorine *Deacon* reaction is lying mainly on the left side (much HCl , little Cl_2). With decreasing flue gas temperature during boiler passage, this reaction should produce more and more Cl_2 until its stop temperature is reached, which was evaluated to be 680°C , [5].

Below this temperature until the boiler exit, the chlorine *Griffin* reaction is consuming more and more of the intermediately formed Cl_2 , depending on the SO_2 concentration, which itself is influenced by the direct SO_2/SO_3 conversion, having been kinetically stopped already at a considerably higher stop temperature of 900°C .

The stop temperature of the direct mercury chlorination itself was evaluated to be 580°C in the studied HWC plants and 480°C respectively in the studied PC-fired wet bottom boiler.

Bromine-based mercury oxidation is governed by a similar, but not exactly corre-

sponding set of global reactions, Figure 9. Even at higher temperatures the bromine *Deacon* reaction is lying far more at the side of free bromine, i.e. the bromine *Deacon* reaction is producing comparatively much more free halogenes (Br_2) than the chlorine *Deacon* reaction is producing Cl_2 . The next completely different step is the *Griffin* reaction. In contrast to chlorine, the bromine *Griffin* reaction is thermodynamically not favoured, at least at temperatures $> 100^\circ\text{C}$, because the *Gibbs* free reaction enthalpy of the bromine *Griffin* reaction is strongly positive, within the whole boiler temperature range. Therefore, SO_2 is not consuming Br_2 during boiler passage or during passage of a high dust SCR-DENOX catalyst bed. There is a further difference to be considered: While Cl_2 is almost insoluble in water and its solubility is even decreasing with growing content of halogenides, Br_2 is easily soluble in water, especially in the presence of halogenides. At temperatures below 100°C the so-called bromine *Bunsen* reaction will be going on (consumption of free bromine by SO_2 under formation of 2HBr and H_2SO_4), Figure 9.

In the stationary estate of a SCR-DENOX both Hg_{met} -adsorption and chemically enhanced Hg^{2+} -desorption are in a dynamic equilibrium.

Instationary mercury oxidation in a SCR-DENOX was already studied in [2] and published in [6]: Hg_{met} -peaks entering the SCR-catalyst bed are not “answered” by corresponding peaks at the SCR-exit: Mercury is leaving the SCR catalyst bed as oxidised mercury Hg_{ion} only and at low, exponentially decreasing concentrations. Thus, mercury oxidation in any SCR-DENOX catalyst bed has to be interpreted as a superposition of Hg_{met} -adsorption and of chemically enhanced Hg^{2+} -desorption (elution). The mercury fluxes as well as the momentary mercury load of the catalyst bed will be permanently adjusted corresponding to the chang-

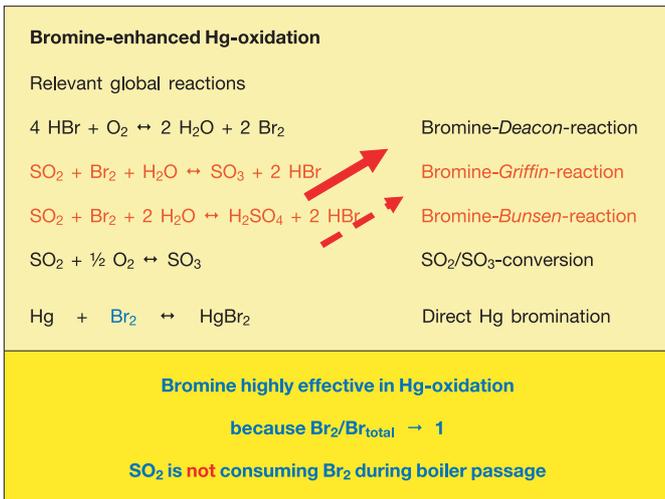


Figure 9. Relevant global reactions of mercury bromination in boiler flue gases.



Figure 10. Storage container of diluted NaBr solution and pumps during test runs at two SFBC plants of Emschergenossenschaft in Bottrop, Germany [5].

ing flue gas concentrations of mercury species and of reactive compounds as SO₂, Cl₂ or Br₂.

Since our in-plant research started in 2000, it soon became known – under high chlorine loads in the raw gas – that SO₂ peaks are inhibiting mercury elution out of a tail-end SCR-DENOX, while Cl₂ peaks are enhancing mercury elution [2]. This fact becomes also (indirectly) visible in Figure 8, showing those artificially induced oscillations of the SO₂ concentration in the boiler raw gas inducing countercurrent oscillations of the Hg_{total} concentration (mainly oxidised mercury) in the effluent flue gas behind the SCR: Higher SO₂ concentrations in the boiler raw gas are inducing better Cl₂ chemisorption in the alkaline scrubber (reducing residual hypochlorides), thus diminishing traces of Cl₂ in the scrubbed flue gas and therefore diminishing also the mercury elution out of the SCR catalyst bed.

Low-cost Technology

The bromine enhanced mercury abatement is a low-cost technology, with respect to the amount of bromine necessary for complete mercury oxidation (Br/Hg > some 300 kg/kg) as well with respect to low installation costs. This may be illustrated by Figures 10 and 11 from those test runs at the two SCCB-plants of Emschergenossenschaft in Bottrop. Figure 10 is showing two (alternatively used) plastic tanks (1000 l) for diluted sodium bromide solution and three small membrane pumps to pump the solution to three dual fluid spaying nozzles, installed in the lower SFBC freeboard region as shown in Figure 11.

Achievable Mercury Removal Efficiencies

Even in flue gases of very high SO₂ concentration a complete mercury bromination is easily attained by adding small amounts of

bromine into the combustion chamber. Thus, the achievable mercury removal efficiency can be > 90 %, provided that the wet or dry gas cleaning system downstream is well designed and well operated, Figure 12.

To minimise the bromine need, good distribution and mixing of the bromine compound with the flue gas is necessary.

Polychlorinated and Polybrominated Dibenzodioxins and – furans

Denovo-synthesis of polychlorinated and polybrominated dioxins and furans (PCDD/F and PBDD/F) were investigated at both SFBC plants in Bottrop and shown to be irrelevant. The measurements were done during bromide addition. The PCDD/F were lying with 0.001 to 0.003 ng TEQ (Nato/CCMS)/Nm³ dry well below 0.1 ng TEQ/Nm³ dry as limiting value of the 17. BImSchV (German emission control act). The corresponding values of PBDD/F were lying with 0.0003 ng TEQ/Nm³ dry near to the detection limit.



Figure 11. Bromide injection into the SFBC freeboard during test runs at SFBC plants of Emschergenossenschaft in Bottrop, Germany [5].

Dry Mercury Adsorption on Carbonaceous and Non-carbonaceous Sorbents

It has been demonstrated by in plant experiments [3], [4] (Figure 14) and by laboratory tests [11], that bromine addition into the combustion chamber increases mercury adsorption at carbonaceous sorbents. The adsorption is enhanced by “internal bromination” of unburned fly ash carbon (UBC) or of injected normal powdered activated carbon (PAC), and specific non-carbonaceous sorbents of high porosity, too.

The adsorption of brominated mercury at ESP fly ashes with relatively high UBC contents was especially studied in 2002 during

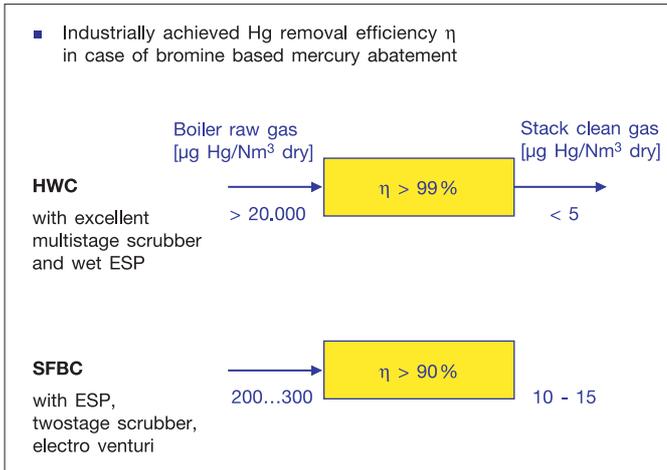


Figure 12. Achievable mercury removal efficiencies.

the test runs at a 100 MW_{therm} coal-fired wet bottom boiler of BAYER AG in Uerdingen, Figure 13. It was demonstrated [4] that the injection of only small amounts of bromine into the combustion chamber (as e.g. hydrogen bromide or as sodium bromide) was sufficient for complete mercury bromination and intensifying the mercury adsorption at the ESP fly ash by the factor 2.5 to 3 in case of high loss on ignition (LOI), Figure 14.

Similar effects on mercury adsorption at the ESP fly ash were observed in December 2003 during the afore mentioned test runs at the SFBC plants of Emschergenossenschaft in Bottrop.

Mercury Oxidation in SCR-DENOX Plants

During those test runs in 2002 at the PC-fired boiler shown in Figure 13, continuous mercury measurements were carried out in front of and behind the wet flue gas desulphurisation

(FGD) as well as behind one of the tail-end DENOX SCRs, i.e. at the stack. These measurements revealed – for the first time, as far as we know – that already tiny amounts of HBr/Br₂ (well below some 1 mg/Nm³ dry) are drastically accelerating mercury oxidation in a SCR catalyst bed, as visible by accelerated mercury elution out of the tail-end SCR-DENOX under high boiler bromide load, Figure 15.

The chlorine *Deacon* equilibrium under low Cl_{total} concentration as in tail-end SCR-DENOXs is lying at the side of HCl, see “HCl excursions behind tail-end SCR-DENOX” as observed and explained in [7]. In contrast to chlorine, the bromine *Deacon* equilibrium is under these conditions lying at the side of Br₂, even in case of very low Br_{total} concentrations, as already shown by *Wieland* [8]. Thus, from HBr in the SCR free bromine is formed oxidising the adsorbed

metallic mercury and promoting the elution of adsorbed HgBr₂.

Laboratory Research on Bromine-Enhanced Mercury Abatement

In early 2002 mercury-related research work was started at the Institute for Environmental Engineering at the Martin-Luther-University Halle-Wittenberg (Campus Merseburg) by *Professor Köser*. The start-up of this research work was possible with financial support of BAYER Industry Services GmbH & Co. oHG. The post-graduate work coached by *Professor Köser* and *Professor Vosteen* is mainly centred around bromine-enhanced mercury abatement and all its aspects in wet and dry flue gas cleaning.

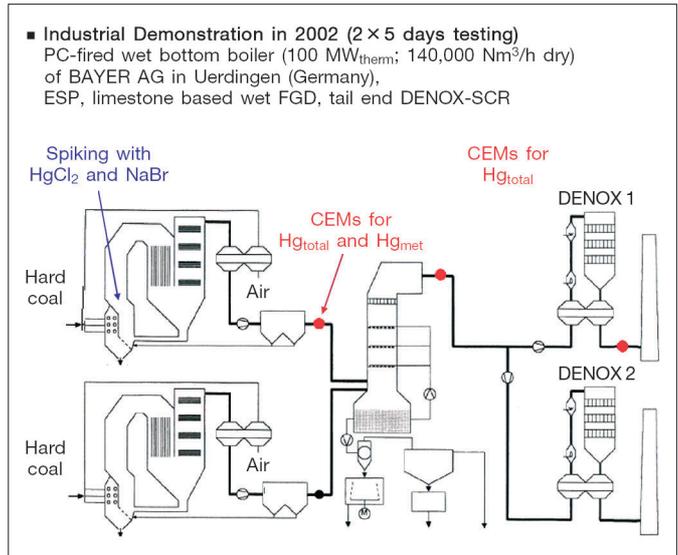


Figure 13. Industrial demonstration of bromine-enhanced mercury abatement in 2002 at a PC-fired (hard coal) wet bottom boiler of BAYER AG in Uerdingen, Germany [4].

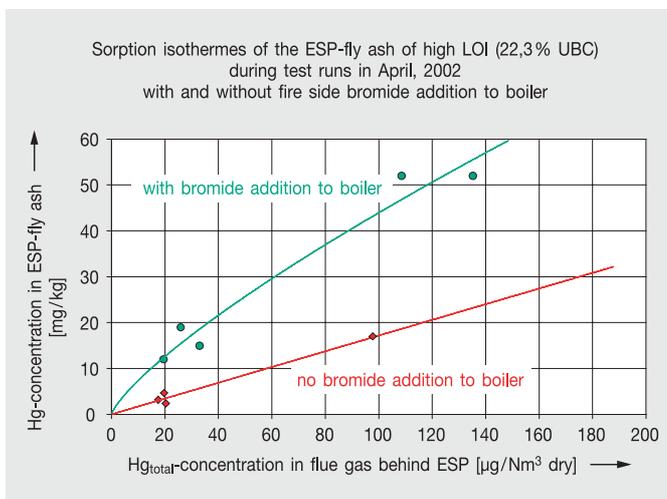


Figure 14. Increase of mercury adsorption at the ESP fly ash (heightening factor 2,5) during a demonstration test run in April 2002 – example with extremely high UBC content to show influence of unburnt carbon.

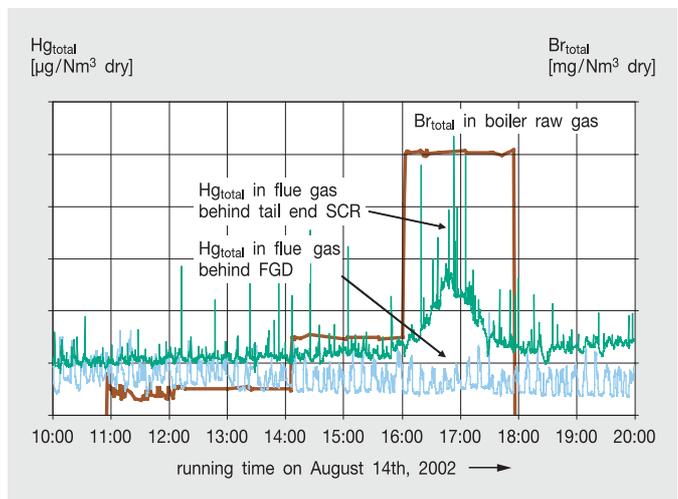


Figure 15. Hg_{total}-concentration in the scrubbed gas in front of tail-end DENOX SCR and in the cleaned gas behind (at the stack) and Br_{total}-concentrations in the boiler raw gas during test run at August 14th, 2002 [4].

In 2004 other companies such as E.ON Engineering (*Professor Gutberlet*) were joining in.

Since 2002 the mercury research group has built quite a lot of test rigs as listed below:

- flue gas chemistry (test rig with high temperature reactor),
- wet flue gas cleaning: mercury complexation by halogenides (test rig),
- wet flue gas cleaning: red-ox-potential and pH, pCl or pBr (test rig),
- dry flue gas cleaning: adsorption at different sorbents (test rig),
- mercury retention and mercury oxidation in tail-end SCR-DENOX (test rig),
- mercury oxidation in high temperature SCR-DENOX (test rig),
- CEM and SCEM artefacts caused by free halogens (test rig); development of a dry mercury converter working at low temperature.

Of considerable interest are new insights into mercury complexation in FGD scrubbers by bromides and their inherent protection against mercury reduction.

Some of the running laboratory research on wet and/or dry mercury abatement was concerned with artifacts in the continuous measuring of total mercury by CEMs or SCEMs with wet or dry gas pre-treatment; a proprietary method to eliminate such artifacts as caused by free halogens was developed and established.

Demonstration Test Runs in the USA

Vosteen Consulting GmbH has been granting an exclusive license to ALSTOM ECS (Environmental Control Systems), Knoxville/TN, for joint exploitation and commercialisation of this technology in North America. ADFA-ES in co-operation with Alstom ECS and others have conducted demonstration test runs under the US-DOE contract No. DE-FC26-03NT41986 at three different sites burning power river basin (PRB) coals, i.e. in the Holcomb Station in Kansas equipped with spray drying absorption and bag filter as air pollution control, in the Laramie River Station U3 equipped with spray drying absorption and ESP and in the Meramec Station U2 equipped with ESP only [13].

In July 2005 Vosteen Consulting GmbH has given a research licence to the Energy and

Power Research Institute, Palo Alto (US EPRI) for demonstration test runs under US patent 6,878,358 at various coal-fired power station boilers of different dry and wet flue gas cleaning design. First short time test runs started in July 2005 to compare mercury chlorination and bromination; in November 2005 a long time demonstration test run under the US-DOE contract No. DE-FC26-03FT41991 ("Large-scale Mercury Control Technology Testing for Lignite-fired Utilities – Oxidation Systems for Wet FGD") applying bromide addition to the coal feed at a 800 MWel unit (TXU's Monticello Steam Electric Station Unit 3) has been run successfully [14]; the evaluation of this test run is still in progress.

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