TUGAS AKHIR

KARAKTERISTIK BATUBARA BLENDING
CANGKANG KELAPA SAWIT

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PROGRAM STUDI TEKNIK KIMIA FAKULTAS TEKNIK

UNIVERSITAS BOSOWA

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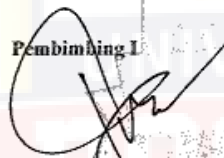### KARAKTERISTIK BATUBARA BLENDING
CANGKANG KELAPA SAWIT

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Telah di pertahankan di depan Dewan Penguji

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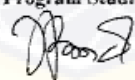
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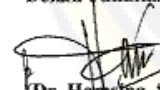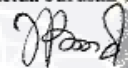Syukur Alhamdulillah, segala puji hanya bagi Allah SWT, karena rahmat dan hidayah-Nya. Penulis akhirnya dapat menyelesaikan penyusunan laporan tugas akhir ini dengan judul " Karakteristik Batubara Blending Cangkang Kelapa Sawit ". Sebagai salah satu syarat guna memperoleh derajat sarjana teknik di Fakultas Teknik , Universitas Bosowa Makassar.

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Makassar, Mei 2017

Salman

DAFTAR ISI

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Simbol	Arti	Satuan
^{o}C	Derajat	Celsius
Hc	Gross Heat of Combustion	Kalori/gr
W	Energi Equivalent (EE)	Kalori
M1	Massa	gr
M2	Massa	gr
M3	Massa	gr
M4	Massa	gr
ad	Air Dry	%
S	Sulfur	%
M	Moisture	%

INTISARI

Tujuan penelitian ini adalah untuk mengukur seberapa besar pengaruh rasio yang di timbulkan apabila batubara di blending dengan cangkang kelapa sawit dan dapat mengetahui peningkatan kualitas yang signifikan apabila batubara di blending dengan cangkang kelapa sawit. Metode yang di gunakan adalah metode fisika, yaitu memblending batubara dengan cangkang kelapa sawit sehingga menjadi produk yang bermanfaat. Sebagai standard acuan analisanya adalah ASTM (American Standard of Testing Material). Hasil penilitian ini dilakukan blendingan dengan 3 variasi yaitu : variasi pertama 25% batubara di blending dengan 75% Cangkang kelapa sawit dengan kadar air 9,72%, kadar abu 5,71%, kadar sulfur 0,18% dan kalori 4701 kalori/gr. Untuk variasi ke dua 50% batubara di blending dengan 50% Cangkang kelapa sawit dengan kadar air 11,28%, kadar abu 5,45%, kadar sulfur 0,33% dan kalori 4977 kalori/gr. Untuk variasi ke tiga 75% batubara di blending dengan 25% Cangkang kelapa sawit. kadar air 11,12%, kadar abu 5,23%, kadar sulfur 0,52% dan kalori 5199 kalori/gr. Berdasarkan penelitian yang telah dilakukan, dapat diambil kesimpulan bahwa Rasio yang di timbulkan apabila batubara di blending dengan cangkang kelapa sawit adalah memberikan nilai sulfur yang signifikan ramah lingkungan, dari penurunannya mulai 0,52 % ke 0,33 % sampai 0,18 %. Kualitas batubara meningkat dengan sulfur semakin rendah, yang ramah lingkungan serta cangkang kelapa sawit sebagai limbah, menjadi maksimal penggunaannya.

Kata kunci : Karakteristik, Blending, Batubara, Cangkang Kelapa Sawit, Standar Acuan

BAB I

PENDAHULUAN

1.1   Latar Belakang

Kelapa sawit merupakan salah satu komoditi unggulan perkebunan di Kalimantan Timur. Produksi tandan buah segar kelapa sawit di Kalimantan Timur pada tahun 2014 mencapai 9.628.072 ton (Badan Pusat Statistik Kalimantan Timur, 2015). Dengan besarnya jumlah produksi kelapa sawit maka berdampak dengan banyaknya limbah yang dihasilkan. Salah satu limbah padat yang dihasilkan dari tandan buah segar kelapa sawit adalah cangkang kelapa sawit. Cangkang dihasilkan sebanyak 7% dari tandan buah segar (Purwaningsih dkk, 2000 dalam Mulia 2007).

Saat ini belum ada pemanfaatan yang maksimal dari cangkang kelapa sawit, karena belum adanya pemanfaatan yang maksimal maka diperlukan pengolahan cangkang kelapa sawit untuk menjadi suatu produk yang bermanfaat dan memiliki nilai ekonomi.

Indonesia sangat kaya akan endapan batubara. eksplorasi selama decade terakhir memperkirakan sekitar 36.633 miliar ton, dimana 67,37 % ada di Sumatra, 32,16 % ada di Kalimantan dan sisanya tersebar Irian Jaya, Jawa dan Sulawesi. Dari seluruh cadangan tersebut, hanya 4.800 miliar ton (13,38 %) dikategorikan sebagai cadangan terukur, sementara sisanya termasuk dalam cadangan terindikasi.

Kegiatan utama dalam eksplorasi dan penambangan difokuskan pada cadangan yang dangkal substansi dari steaming coal sub-butiminous dan bitominous yang terletak di Sumatra, Kalimantan Timur dan Selatan.

Batubara ada di sepanjang Indonesia dangan cadangan utama terletak di Sumatra, Kalimantan Timur dan Selatan. Batubara umumnya ditemukan di lembah sungai sediment dekat dengan permukaan memungkinkan dilakukan penambangan secara terbuka dengan lapisan tanah penutup yang tipis sampai pada ketebalan batubara mencapai 70 meter.

Kualitas batubara Indonesia yang di produksi pada umumnya dikelompokan sub bitiminous yang di rincikan nilai kalori yang tinggi (5.700 – 7.217 kcal/kg), volatile matter yang tinggi (37% - 42%), abu yang rendah (3% - 12%) dan belerang yang rendah (0,10% - 0.85%). Sifat fisik dan kimia batubara bervariasi dari suatu tempat ketempat lainnya.

Batubara Indonesia yang di temukan berdasarkan kualifikasinya dapat dikelompokkan sebagai *lignite* (58.83%), *sub bitiminous* (26,63%), *bituminous* (14,38%) dan *antrasit* (0.36%). (chris 2003).

Cangkang kelapa sawit mengandung kadar arang (20,5%), kadar air (7,8%), kadar abu (2,2%) dan zat mudah menguap (69,5%) (Hartanto dan Ratnawati, 2010). Salah satu potensi dari cangkang kelapa sawit dapat di blending dengan batubara sebagai bahan bakar untuk power station pembangkit tenaga listrik, yang mana cangkang kelapa sawit yang tadinya limbah menjadi bermanfaat dan batubara menjadi maksimal pemanfaatannya dengan adanya proses blendingan ini.

Kelebihan dari cangkang kelapa sawit dibandingkan dengan batu bara adalah cangkang kelapa sawit lebih ramah bagi lingkungan dan orang sekitar. Unsur batubara mengandung sulfur dan nitrogen sehingga pembuangan uap dari boiler akan menggangu kesehatan masyarakat. Saat ini pemanfaatan cangkang sawit diberbagai industri pengolahan minyak CPO masih belum digunakkan sepenuhnya, sehingga masih meninggalkan residu, yang akhirnya cangkang ini dijual mentah ke pasaran (Purba, 2004).

1.2 Rumusan Masalah

Berdasarkan uraian latar belakang, bahwa untuk mengetahui karakteristik batubara di blending dengan cangkang kelapa sawit maka di perlukan beberapa langkah antara lain :

1.2.2 Seberapa besar pengaruh rasio terhadap karakteristik batubara yang di blending dengan cangkang kelapa sawit.

1.2.3 Bagaimana kualitas batubara jika di blending dengan cangkang kelapa sawit.

1.3 Tujuan Penelitian

Pelaksanaan Penelitian ini merupakan program kurikulum yang bertujuan:

1.3.1 Menetukan kondisi optimun perbandingan rasio yang ditimbulkan apabila batubara di blending dengan cangkang kelapa sawit .

1.3.2 Menentukan peningkatan kualitas yang signifikan apabila batubara di blending dengan cangkang kelapa sawit.

1.4 Manfaat

Manfaat dari penelitian ini adalah sebagai alternatif pemanfaatan limbah cangkang kelapa sawit, agar memiliki nilai ekonomis yang lebih tinggi dan batubara yang di gunakan sebagai blendingannya menjadi batubara yang berkualitas lebih baik.
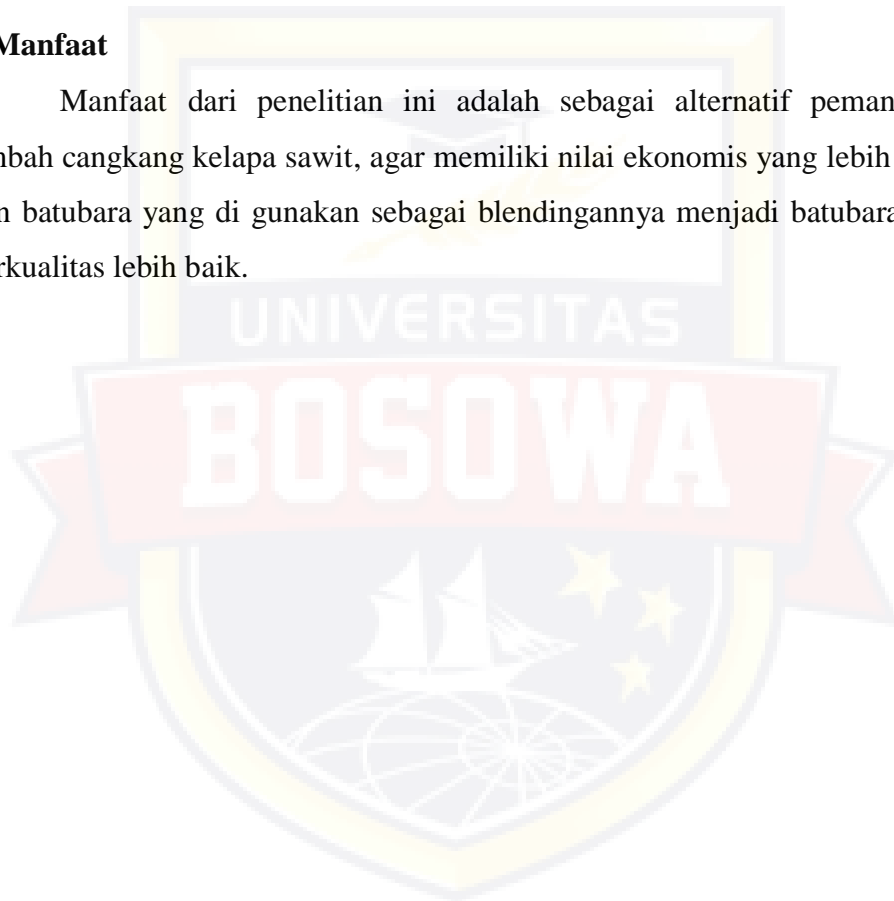
BAB II

TINJAUAN PUSTAKA

2.1 Batubara

Dalam mendefinisikan batubara, harus ditinjau dari beberapa aspek antara sifat fisiknya dan asal kejadian dan pemanfaatannya. Untuk memberikan gambaran mengenai pengertian batubara secara umum oleh beberapa penemu dapat diuraikan dibawah ini:

Menurut *"The International Hand Book of Coal Petrography"* dalam penerbitannya yang kedua pada tahun 1963 memberikan pengertian batubara yaitu "Batubara adalah batuan sedimen yang mudah terbakar, terbentuk dari sisa-sisa tanaman dalam variasi tingkat pengawetan, diikuti oleh proses kompaksi dan terkubur dalam cekungan diawali pada kedalaman yang tidak terlalu dangkal. Cekungan-cekungan ini pada garis besarnya dibagi atas cekungan *lignite (intra continental)* dan cekungan paralis yang berhubungan dengan air laut, segera setelah lapisan-lapisan dasar turun terus menerus, sisa tanaman yang terkubur tersebut dipengaruhi oleh proses normal *metamorfosis* terutama oleh temperatur dan tekanan".

Menurut *"Thiessen (1947)"* menemukan beberapa kejanggalan batubara, sehingga beliau mendefinisikan batubara sebagai berikut:

"Batubara adalah suatu benda padat yang kompleks, terdiri dari bermacam unsur-unsur yang mewakili banyak komponen kimia, dimana hanya sedikit dari komponen kimia tersebut dapat diketahui. Pada umumnya benda padat tersebut homogen, tetapi hampir semua berasal dari sisa-sisa tanaman yang kompleks dengan sendirinya akan berkomposisi dengan sejumlah komponen kimia dalam perbandingan yang bervariasi. Jadi dapat disimpulkan bahwa pengertian dari Thiessen terhadap batubara adalah suatu benda padat organik yang memiliki komposisi kimia yang sangat rumit".

Menurut "Achmad Prijono, dkk Pada Tahun 1992" mengemukakan bahwa batubara adalah bahan bakar hydro-karbon padat yang terbentuk dari

tumbuhan dalam lingkungan bebas oksigen dan terkena pengaruh panas serta tekanan yang berlangsung lama sekali.

Dari ketiga definisi yang telah di uraikan di atas, maka dapat diambil suatu rangkuman pengertian batubara sebagai berikut:

" *Batubara adalah suatu karbon berlapis yang terbentuk oleh akumulasi sisa–sisa tanaman bersama hasil komposisinya yang terawetkan dalam lapisan sediment dan menjadi kaya akan unsur karbon dengan adanya proses diagenesis*".

2.1.1 Proses Pembentukan Batubara

Perkembangan evolusi tanaman yang telah merubah komposisi tanaman pembentuk gambut sejalan dengan waktu sangat terasa penting pengaruhnya terhadap formasi batubara.

Jenis tanaman pada umur karbon tua lebih sedikit jika dibandingkan dengan jenis tanaman yang lebih muda, begitu juga dengan komposisi kimianya sehingga akan menghasilkan batubara yang berbeda pula misalnya tanaman bambu yang berumur tersier lebih peka terhadap proses pembusukan sehingga batubara yang dihasilkan lebih kaya akan komponen berminyak dan resin. Sebagai kesimpulan dapat diutarakan bahwa batubara yang berumur tersier akan berbeda dengan batubara yang berumur paleosoik, karena tanaman pembentuknya berbeda seperti :

a. Iklim

Temperatur dan tekanan memegang peranan penting dalam proses terbentuknya batubara dimana kaitannya dengan proses kecepatan pertumbuhan tanaman, jenis kehidupan tanaman dan kecepatan pembusukan.

Iklim sangat tergantung dengan posisi geotektonik sedangkan topografi purba hanya berhubungan langsung dengan cekungan pembentukan batubara. Jenis iklim tropis, panas dan basah serta sub tropis merupakan iklim yang sesuai perkembangan hutan-hutan rawa.

Lapisan batubara dengan gambut modern (biasa terendapkan di dalam delta) dapat terbentuk dengan tingkat kecepatan penimbunan yang lebih cepat dari tingkat kecepatan pembusukan.

b. *Posisi Geografis*

Garis-garis lintang purba dan ketinggiannya mempunyai pengaruh penting dalam penentuan keadaan iklim, terutama temperatur.Rawa-rawa paralis yang sangat penting untuk pembentukan pada permukaan laut walaupun batubara sebenarnya dapat terbentuk pada ketinggian yang berlainan. Jadi dengan bertambahnya ketinggian, berarti temperatur akan lebih rendah dan merupakan faktor pembatas dalam pembentukan formasi batubara.

c. *Proses Biokimia*

Setelah tanaman mati, proses degradasi biokimia banyak berperan. Bila tanaman terakumulasi dalam lingkungan rawa atau payau, mereka akan menjadi jenuh air, proses pembusukan "decay" akan tejadi oleh kerja mikrobiologi atau bakteri anaerobik. Bakteri ini bekerja dalam suasana tanpa oksigen dan mempunyai kemampuan yang sama terhadap bakteri aerobik. Aktifitas mikrobiologi yang berupa bakteri dan fungsi tersebut pertama-tama menghancurkan bagian yang lunak seperti sellulosa, protoplasma dan pati. Sedangkan bagian yang lebih keras seperti lilin, damar, kulit kayu akan tertinggal.

Aktifitas mikrobiologi dalam pembentukan batubara tergantung pada jumlah sirkulasi air, suplay oksigen dan perkembangan racun (hasil samping dari aktivitas mikrobiologi). Bila salah satu dari faktor tersebut tidak berimbang, maka aktivitas mikrobiologi akan terganggu. Aktivitas mikrobiologi terutama bakteri yang lebih cenderung dipermukaan.

d. *Proses Kimia Fisik*

Setelah mengalami proses biokomia maka selanjutnya dalam proses pembetukan batubara mengalami proses kimia fisika yang merupakan

tingkat penimbunan atau penyuburan. Dalam tingkat ini proses degradasi biokimia tidak berperan lagi, tetapi lebih didominir oleh proses dinamo kimia atau kimia fisik. Proses inilah yang menyebabkan perubahan gambut menjadi batubara dalam berbagai derajat.

Selama proses ini terjadi pengurangan air lembab, oksigen dan zat terbang, sulfur dan kandungan abunya dan bertambahnya presentase karbon padat. Faktor dominan utama dalam pembentukan batubara adalah tekanan temperatur dan waktu. Selain itu dalam pembentukan batubara terdapat beberapa faktor yang menentukan antara lain sumber material, tempat, iklim proses dekomposisi,dan material.

Proses Pembentukan batubara mencakup beberapa proses sebagai berikut:

2.1.1.1 Pembusukan

Proses pembusukan tanaman terjadi akibat adanya aktifitas dari bakteri anaerob (degradasi anaerob). Bakteri ini bekerja dalam suasana tanpa oksigen dengan menghancurkan bagian yang lunak dari tumbuhan seperti sellulosa, protoplasma, dan pati. Aktifitas mikrobiologi dalam pembentukan batubara tergantung pada jumlah dan sirkulasi air, temperatur air, supplai oksigen, perkembangan racun (hasil samping dari aktifitas mikrobiologi). Bila salah satu dari faktor tersebut tidak berimbang, maka aktifitas mikroboiologi tidak akan terjadi.

2.1.1.2 Pengendapan

Proses dimana material halus hasil pembusukan terakumulasi dan mengendap membentuk lapisan gambut. Proses ini biasa terjadi di lingkungan berair misalnya rawa – rawa.

2.1.1.3 Dekomposisi

Proses dimana lapisan gambut tersebut di atas akan mengalami perubahan berdasarkan proses biokimia yang berakibat keluarnya air dan sebagian akan hilang dalam bentuk carbon dioksida, carbon monoksida, dan metan. Proses ini yang menyebabkan perubahan

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gambut menjadi batubara dalam berbagai derajat.

Selama proses ini, terjadi penguraian air lembab, oksigen dan zat terbang serta bertambahnya persentase carbon padat, Sulfur dan kandungan abunya. Proses dekomposisi tumbuhan menjadi lignit dapat dituliskan dalam reaksi sebagai berikut:

$5(C_6H_{10}O_5) \longrightarrow C_{20}H_{22}O_4 + 3CH_4 + 8H_2O + 6CO_2 + CO$

(selulosa)   *(lignit) (metan)*
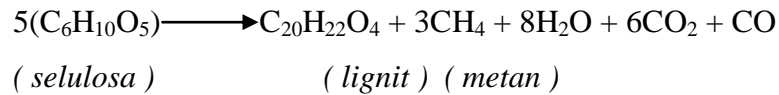
2.1.1.4 Geotektonik

Lapisan gambut yang ada akan terkompaksi oleh gaya tektonik, dan kemudian dalam fase selanjutnya akan mengalami perlipatan dan patahan. Selain itu gaya tektonik dapat menimbulkan adanya intrusi/terobosan dari magma. yang mengubah batubara *low grade* menjadi *high grade*. Dengan adanya tektonik setting tertentu, maka zona batubara yang terbentuk dapat berubah dari lingkungan air menjadi lingkungan darat.

Secara sederhana, batubara merupakan suatu endapan yang berasal dari tumbuh – tumbuhan yang mengalami proses penghancuran karena aktifitas bakteri pengendapan, penumpukan serta pemadatan. Karena pengaruh proses geologi, yaitu dengan adanya peningkatan tekanan maupun temperatur, maka akan terbentuklah batubara.

2.1.2 Komposisi Batubara

Penilaian baik tidaknya atau tinggi rendahnya kualitas batubara ditentukan oleh komposisi kimia dan fisika yang dimiliki oleh batubara. Komposisi kimia dari batubara pada umumnya sebagian adalah carbon (40% - 80%) dan sebagian kecil adalah unsur pengikut seperti unsur *oksigen (O), hidrogen (H), sulfur (S), natrium (Na), kalsium (Ca), kalium (K), ferrum (Fe), almunium (Al), magnesium (Mg), silikon (Si)* dan lain sebagainya. Unsur H, O, N, dan S terdapat dalam batubara biasanya dalam bentuk senyawa seperti H_2O, CH_2, SO, NO_2 sedangkan bentuk lain

berbetuk oksida seperti Al_2O_3, MgO, K_2O, Fe_2O_3, Na_2O, SiO_2 dan sebagainya. Pada dasarnya terdapat dua jenis material yang membentuk batubara, yakni:

a. *Combustible Matter (Bahan Dapat Terbakar)*

Bahan dapat terbakar yaitu bahan atau mineral yang dapat teroksidasi oleh oksigen menghasilkan kalor. Materi tersebut umumnya terdiri dari:

➢ Karbon padat (*fixed carbon)*, yang jika terbakar membentuk kokas, kokas mempunyai kandungan karbon + 80 %

➢ Senyawa hidrokarbon

➢ Senyawa sulfur, dan

➢ Senyawa hydrogen, serta beberapa senyawa lainnya dalam jumlah kecil.

b. *Non Combustible Matter (Bahan yang Tidak Dapat Terbakar)*

Bahan yang tidak mudah terbakar yaitu bahan atau mineral yang tidak dapat terbakar/teroksidasi oleh oksigen. Material tersebut umumnya terdiri dari senyawa-sanyawa anorganik (SiO_2, Al_2O_3, TiO_2, MnO_2, CaO, MgO, Na_2O, K_2O dan senyawa-senyawa logam lain dalam jumlah kecil) yang akan membentuk abu dalam batubara. Bahan yang tidak dapat terbakar ini umumnya tidak diinginkan keberadaannya karena akan mengurangi kalori batubara.

2.1.3 Karakteristik Batubara

2.1.3.1 Sifat Fisik

Sifat fisik batubara tergantung kepada unsur kimia yang membentuk batubara tersebut, semua sifat fisik yang dikemukakan di bawah ini mempunyai hubungan satu sama lainnya :

a. Berat jenis, batubara juga sangat bergantung kepada jumlah dan jenis mineral yang dikandung abu dan porositasinya, berat jenis yang rendah menyebabkan sifat pembakaran yang baik. Penentuan berat jenis batubara digunakan sebagai data geologi untuk mengetahui

apakah batubara tersebut layak ditambang dengan memperhitungkan faktor komersial.

b. Kekerasan, kekerasan berkaitan dengan struktur batubara. Keras atau lemahnya batubara juga tergantung kepada komposisi dan jenis batubaranya.

c. Berdasarkan warna, batubara dibedakan menjadi dua jenis, yaitu batubara cerah dan batubara kusam.

2.1.3.2 Sifat Kimia

Sifat kimia dari batubara berhubungan langsung dengan senyawa penyusunan dari batubara tersebut, baik senyawa organik ataupun senyawa anorganik.

2.1.4 Klasifikasi Batubara

Batubara dengan mutu yang lebih tinggi umumnya lebih keras dan kuat dan seringkali berwarna hitam cemerlang seperti kaca. Batubara dengan mutu yang lebih tinggi memiliki kandungan karbon yang lebih banyak, tingkat kelembaban yang lebih rendah dan menghasilkan energi yang lebih banyak.

Adapun tingkatan –tingkatan dari batubara antara lain :

a. Peat, Peat terbentuk dari rontokan daun yang berubah menjadi batu bara yang mengandung sisa tanaman yang tidak benar-benar busuk dan memadat. Dibedakan dengan tingkat rendah batubara terendah oleh adanya selulosa bebas.

b. Lignite, batubara tingkat rendah yang mengandung gross calorific value kurang dari 19,3 Mj/kg. Batubara ini memiliki ciri-ciri sebagai berikut: moisture dan volatile matter yang tinggi. Lignite ada yang hitam, coklat dan brown coal, nyala berasap, nilai kalori rendah, mudah terbakar.

c. Sub Bituminous, batubara ditandai dengan ciri-ciri moisture yang cukup tinggi dan batu bara jenis ini banyak digunakan untuk industri semen.

d. Bituminous, batubara jenis ini mempunyai ciri-ciri warnanya lebih

hitam, nilai kalor tinggi, kadar air rendah dan mudah terbakar.

e. Antrasit, batubara tingkat tinggi yang memiliki kilapan metal dan kandungan volatile matter yang rendah, kadar Carbon tinggi dan susah terbakar. Antrasit juga oleh para ilmuan dikatakan sebagai batubara yang paling tua umurnya, dibandingkan dengan jenis batubara yang lain.

2.1.5 Pemanfaatan Batubara

Secara umum penggunaan batubara dapat dikelompokkan menjadi tiga, sebagai berikut:

a. *Sebagai Bahan Bakar Langsung*

Batubara dapat digunakan secara langsung dalam bentuk padatan tanpa melalui pengolahan, misalnya digunakan sebagai bahan bakar pada ketel uap, pabrik semen dan pada industri – industri kecil.

b. *Sebagai Bahan Bakar Tak Langsung*

Batubara terlebih dahulu diproses sebelum digunakan menjadi bentuk lain misalnya sebagai sumber energi, proses tersebut diantaranya adalah.

Basifikasi	:	Diproses sebagai bahan bakar gas.
Pencairan	:	Diproses sebagai bahan bakar minyak sintetis.
Karbonisasi	:	Diproses sebagai kokas atau semi kokas yang digerakkan dalam bentuk bongkahan/briket untuk bahan bakar industri atau rumah tangga.
Suspensi	:	Dengan membuat suspensi batubara dalam air, diperoleh coal water fuel yang mempunyai sifat mirip dengan bahan bakar minyak.

c. *Bukan Sebagai Bahan Bakar*

Selain sebagai bahan bakar langsung dan tak langsung, batubara juga dimanfaatkan pada berbagai industri yang menggunakannya bukan sebagai bahan bakar antara lain: sebagai bahan baku industri petrokimia, sebagai reduktor pada peleburan biji timah dan besi, sebagai elektroda, karbon aktif dan lain-lain.

2.1.6 Sampling Batubara

Sampling dari batubara merupakan kegiatan yang kompleks dan sulit, dari pengiriman batubara. Istilah "representatif" berarti sepanjang menyangkut tujuan semua dokumen yang dikeluarkan oleh berbagai organisasi standar mengenai sampling adalah dokumen untuk jangka panjang. Oleh karena itu penting bahwa segala kegiatan sampling ditujukan oleh personil yang kompeten dan terlatih dengan mengikuti standar yang cocok secara tepat.

Sampling merupakan pengambilan sejumlah kecil sampel batubara yang refpresentatif analisa dan testing sampel dalam jumlah sedikit dapat digunakan untuk mewakili jumlah yang lebih besar.

Sampling yang dilakukan pada internal quality control adalah untuk memastikan bahwa produk tersebut sesuai dengan spesifikasi yang ditentukan. Hal ini juga berlaku untuk quality eksternal dimana sutu perusahaan inspeksi yang independent melaksanakan sampling dan pengujian untuk sertifikasi pengapalan batu bara.

Pentingnya sampling, dapat dilihat dibawah ini :

Kesalahan hasil analisa dapat disebabkan oleh:

➢ 80 % dari sampling

➢ 15 % dari preparasi sample

➢ 5 % dari analisa laboratorium

2.1.7 Preparasi dan Analisa Batubara

2.1.7.1 Preparasi Sample

Preparasi sample adalah pengurangan massa dan ukuran dari gross sample sampai pada massa dan ukuran yang cocok untuk analisa di laboratorium. Preparasi sample untuk penentuan moisture dan general analisis biasanya mencakup pembagian dan pengurangan. Hal ini juga mencakup mixing, pengeringan udara dan equalize.

2.1.7.2 Analisa Sample

Metode analisa setiap parameter batubara dijelaskan dalam metode standar, metode standar yang umum digunakan yaitu *ISO (Internasional Standart Organizations), ASTM (American Society for Testing Materials), BS (British Standar)* dan *AS (Australian Standars)* dimana analisa batubara terdiri dari beberapa analisa umum.

2.1.7.2.1 Analisa Proksimat

Analisa proksimat adalah analisa kimia pendahuluan pada sample batubara untuk mengetahui kualitas batubara dengan mengukur parameter – parameter yang dinyatakan dalam persen. Di mana analisis Proksimat ini terdiri dari: *Moisture in the Analysis Sampel, Ash Content, Volatile Matter, dan Fixed Carbon.* Analisis proksimat merupakan analisa awal dalam batubara untuk memenuhi permintaan konsumen. Adapun beberapa analisa proksimat yaitu:

a. *Moisture in the Analysis Sample (MAS)*

Moisture in the Analysis Sample yaitu kandungan air yang terdapat dalam batubara pada saat diperiksa atau pada saat telah dikeringkan dengan udara.Besar kecilnya *MAS* ini dipengaruhi oleh peringkat batubara, dan temperature pada saat batubara dianalisa, dan juga berpengaruh pada preparasi sample sebelum *MAS* dianalisa.

Moisture adalah kandungan air yang terikat atau yang terdapat dalam batubara pada kondisi normal. Kandungan air berhubungan erat dengan derajat sample batubara asal. Untuk menentukan kadar air dalam batubara dapat dilakukan dengan dua metode yaitu standar *ASTM*, dengan menggunakan udara kering dan standar *ISO*, dengan menggunakan gas nitrogen. Kandungan air dalam sample dapat didefinisikan sebagai persentase berat yang hilang jika sample batubara dipanaskan pada kondisi temperatur standar yakni pada suhu $105^{o}C$.

Kandungan air yang tinggi dalam batubara dapat menyebabkan penurunan mutu batubara sebab:

➤ Menurunkan nilai kalor batubara.

➤ Menurunkan titik nyala.

➤ Memperlambat proses pembakaran.

b. *Ash Content (Kandungan Abu)*

Di dalam analisis batubara, abu didefinisikan sebagai sisa pembakaran yang tinggal jika batubara dipijarkan Material pembentuk abu yang terbentuk dalam batubara terdiri dari dua macam yaitu *inherent* dan *extraneous* (tidak berhubungan dengan materi pembentuk batubara).

Kadar abu dari batubara penting diketahui sebab:

➤ Kadar abu memberikan indikasi dasar terhadap kekotoran batubara sehingga dapat dipakai sebagai dasar untuk perencanaan kelayakan pembakaran/tanur.

➤ Kadar abu mencerminkan banyaknya mineral dalam batubara dan secara tidak langsung mencerminkan jumlah nilai kalor dari batubara. Bila kadar abu tinggi maka nilai kalor rendah.

2.1.7.2.2 *Gross* **Calorific Value (Nilai kalori)**

Nilai Kalori adalah jumlah panas yang dihasilkan oleh suatu bahan bakar yang dibakar pada kondisi yang terkendali. *Gross Calorific Value* adalah jumlah unit panas yang dikeluarkan per unit bahan yang dibakar dengan oksigen di bawah kondisi standar. Nilai kalori biasanya dinyatakan dalam satuan cal/g. Nilai kalori dari sampel batubara ditetapkan dengan cara membakar sampel dalam lingkungan berisi gas oksigen dengan tekanan 30 atm, Panas yang dilepaskan oleh pembakaran setimbang dengan nilai kalori sampel.

2.2 Cangkang Kelapa Sawit

Kelapa sawit *(Elaeis Guineensis Jacq)* merupakan tanaman penghasil utama minyak nabati yang berasal dari Afrika Barat. Tanaman ini pertama kali diperkenalkan di Indonesia oleh pemerintah Hindia Belanda tahun 1848. Saat itu ada 4 batang bibit kelapa sawit yang ditanam di Kebun Raya Bogor (Botanical Garden), dua berasal dari Bourbon (Mauritius), dua lainnya berasal dari Hortus Botanicus, Amsterdam (Belanda). Beberapa pohon kelapa sawit yang ditanam di Kebun Raya Bogor hingga tahun 2014 masih hidup dengan ketinggian sekitar 12 m. Tanaman tersebut merupakan kelapa sawit tertua di Asia Tenggara yang berasal dari Afrika (Pardamean, 2014).

Kelapa sawit dapat tumbuh dengan baik di daerah tropis (15° LU - 15° LS) dengan ketinggian tempat 0 - 500 m dari permukaan laut dengan kelembaban 80% - 90%. Kelapa sawit membutuhkan iklim dengan curah hujan stabil 2000 – 2500 mm setahun yaitu daerah yang tidak tergenang air saat hujan dan tidak kekeringan saat kemarau. Pola curah hujan tahunan mempengaruhi prilaku pembungaan dan produksi buah sawit (Wikipedia, 2015).

Industri kelapa sawit merupakan salah satu industri strategis yang bergerak pada sektor pertanian yang banyak berkembang di negara-negara tropis seperti Indonesia, Malaysia, dan Thailand. Hasilnya biasa digunakan sebagai bahan dasar industri seperti industri makanan, komestika dan industri sabun. Perkembangan industri kelapa sawit saat ini sangat pesat, dimana terjadi peningkatan jumlah produksi kelapa sawit seiring meningkatnya kebutuhan masyarakat. Limbah yang dihasilkan dari proses pengolahan minyak kelapa sawit adalah limbah padat, cair dan gas (Agustina, 2006).

Limbah padat yang dihasilkan antara lain tandan kosong, cangkang/fiber, abu boiler, solid decanter, sampah loading ramp dan shell. Sedangkan limbah cair yang dihasilkan dari kegiatan industri pengolahan

minyak kelapa sawit merupakan sisa dari proses pembuatan minyak kelapa sawit yang berbentuk cair. Air limbah hasil samping dari pengolahan kelapa sawit sangat banyak mengandung bahan organik dan dapat mencemari lingkungan bila langsung dibuang ke perairan (Pardamean, 2014).

Fauzi et all. (2014) mengatakan bahwa jumlah limbah cair yang dihasilkan oleh Pabrik Minyak Kelapa Sawit (PMKS) berkisar antara 600 - 700 liter/ton. Limbah yang dihasilkan oleh pabrik pengolahan kelapa sawit dapat mencemari lingkungan, menjadi racun, dan lain-lain. Oleh karena itu, kita harus memanfaatkan limbah tersebut ke lapangan.

Cangkang sawit yang awalnya dari tempurung kelapa sawit, merupakan bagian paling keras pada komponen yang terdapat pada kelapa sawit. Saat ini pemanfaatan cangkang kelapa sawit di berbagai industri pengolahan minyak CPO belum begitu maksimal. Cangkang memiliki kegunaan sebagai bahan bahan arang, bahan bakar untuk boiler (Purba, 2004).

Kelapa sawit adalah salah satu komoditi andalan Indonesia yang perkembangannya demikian pesat. Selain produksi minyak kelapa sawit yang tinggi, produk samping atau limbah pabrik kelapa sawit juga tinggi. Dengan kondisi yang semacam itu sebenarnya banyak sekali manfaat yang dapat diperoleh dari pemanfaatan cangkang sawit tersebut. Salah satunya apabila dilakukan pirolisis terhadap cangkang sawit tersebut akan diperoleh rendemen berupa asap cair yang dapat digunakan sebagai biopreservatif pengganti preservatif kimia, arang maupun tar.

Kelapa sawit memiliki jenis berdasarkan tebal tipisnya cangkang (Setyamidjaja, 2006), yaitu :

2.2.1 Tipe Dura

Tipe ini memiliki ciri-ciri daging buah (mesocarp) tipis, cangkang (endocarp) tebal (2-8 mm), inti (endospern) besar dan tidak terdapat cincin

16

serabut. Presentase daging buah 35-60% dengan rendemen minyak 17%-18%.

2.2.2 Tipe Pisifera

Tipe ini memiliki ciri-ciri daging buahnya tebal, tidak mempunyai cangkang, tetapi terdapat cincin serabut yang mengelilingi inti. Intinya kecil sekali bila dibandingkan dengan tipe Dura ataupun Tenera. Perbandingan daging buah terhadap buahnya tinggi dan kandungan minyaknya tinggi. Bunga kelapa sawit tipe Pisefera biasanya steril. Kelapa sawit tipe ini hanya dipakai sebagai "pohon bapak" dalam persilangan dengan tipe *Dura/Deli Dura*.

2.2.3 Tipe Tenera

Tipe ini merupakan hasil silang antara tipe *Dura* dan *Pisiera*. Sifat tipe *Tenera* merupakan kombinasi sifat khas dari kedua induknya. Tipe ini mempuyai tebal cangkang 0,5-4 mm, mempunyai cincin serabut walaupun tidak sebanyak seperti Pisifera, sedangkan intinya kecil. Perbandingan daging buah terhadap buah 60%-90%, rendemen minyak 22%-24%.

Cangkang kelapa sawit terdiri atas *hemiselulosa* (31,70%), *selulosa* (32,53%), lignin (20,09%), *lemak* (5,33%) dan *protein* (4,45%) (Irawadi, 1991 dalam Habibati, 2010). *Hemiselulosa* termasuk jenis polisakarida berantai pendek serta mempunyai berat molekul kecil dibanding dengan selulosa dan banyak terdapat pada kayu lunak. *Hemiselulosa* bersifat tidak *kristalin*, tetapi sangat bercabang dengan derajat polimerisasi jauh lebih rendah daripada selulosa. Pada kayu, *hemiselulosa* berfungsi sebagai bahan pendukung dalam dinding sel. Penyusun hemiselulosa terdiri dari *pentosan* ($C_5H_8O_4$) dan *heksosan* ($C_6H_{10}O_5$) (Sari, 2015).

Cangkang buah kelapa sawit dapat dimanfaatkan sebagai arang aktif. Arang aktif dimanfaatkan oleh berbagai industri, antara lain industri minyak, karet, gula dan farmasi. Selain itu tempurung kelapa sawit

digunakan hanya sebagai bahan bakar pembangkit tenaga uap dan bahan pengeras jalan (Fauzi dkk., 2002 dalam Zunipar, 2015).

2.2.4 Asal-usul cangkang kelapa sawit.

Cangkang kelapa sawit yang awalnya dari tempurung kelapa sawit, merupakan bagian paling keras pada komponen yang terdapat pada kelapa sawit. Saat ini pemanfaatan cangkang kelapa sawit di berbagai industri pengolahan minyak CPO belum begitu maksimal. Cangkang memiliki kegunaan sebagai bahan bahan arang, bahan bakar untuk boiler (Purba,2004).

Kelebihan dari cangkang kelapa sawit dibandingkan dengan batubara adalah cangkang kelapa sawit lebih ramah bagi lingkungan dan orang sekitar. Unsur batubara mengandung sulfur dan nitrogen sehingga pembuangan uap dari boiler akan menggangu kesehatan masyarakat. Saat ini pemanfaatan cangkang sawit diberbagai industri pengolahan minyak CPO masih belum digunakkan sepenuhnya, sehingga masih meninggalkan residu, yang akhirnya cangkang ini dijual mentah ke pasaran (Purba, 2004).

2.2.5 Cangkang Kelapa Sawit Sebagai Bahan Aktif

Tempurung kelapa sawit merupakan salah satu limbah pengolahan minyak kelapa sawit yang cukup besar dapat mencapai 60 % dari produksi minyak. Arang aktif juga dapat dimanfaatkan oleh berbagai industri seperti industri minyak, karet, gula dan farmasi. Selama ini tempurung kelapa sawit digunakan sebagai bahan bakar pembangkit uap dan pengeras jalan.

Arang aktif dapat dibuat melalui proses karbonasi pada suhu 550°C selama kurang lebih 3 jam. Karakteristik arang aktif yang dihasilkan melalui proses tersebut memenuhi standar industri Indonesia, kecuali untuk kadar abu. Tingkat keaktifan arang cukup tinggi. Hal ini terlihat dari   daya serap larutan ionnya sebesar 28,9 %.

2.2.6 Dampak Limbah Industri Kelapa Sawit

Peningkatan produksi dan konsumsi dunia terhadap minyak sawit secara langsung dapat meningkatkan dampak negatif terhadap lingkungan. Pada proses produksi minyak sawit limbah berwujud padat, cair dan gas yang dihasilkan dari berbagai stasiun kerja dari pabrik. Setiap ton TBS yang dihasilkan diolah menjadi efluen sebanyak 600 liter. Limbah tersebut berdampak negatif terhadap lingkungan jika tidak dimanfaatkan dengan baik. Sekarang ini mulai dikenal pengolahan lingkungan yang bersifat pencegahan terhadap sumber-sumber dihasilkan limbah, seperti eco-efficient, pollution prevention, water minimization atau source reduction. *United Nation Environment Programmer (UNEP)* menggunakan istilah cleaner production atau produksi bersih sebagai upaya preventif dan integrasi yang dilaksanakan secara berkesinambungan terhadap proses dan jasa untuk meningkatkan efisiensi dan mengurangi resiko terhadap manusia dan lingkungan.

Limbah dari industri dapat membahayakan kesehatan manusia karena merupakan sumber penyakit *(sebagai vehicle)*. Limbah industri dapat merugikan dari segi ekonomi karena dapat menimbulkan kerusakan pada benda/bangunan, tanaman, peternakan dan dapat merusak bahkan membunuh kehidupan yang ada didalam air seperti ikan dan binatang peliharaan serta keindahan alam lainnya. (Rusmery, T. 2009).

BAB III

METODE PENELITIAN

3.1 Waktu dan Tempat Penelitian

Penelitian ini dimulai pada semester genap bulan februari hingga april tahun 2017. Penelitian ini akan dilaksanakan di laboratorium PT. SUCOFINDO (persero) Cabang Samarinda. Bahan baku di peroleh dari aktifasi sampel yang masuk ke laboratorium PT. SUCOFINDO (persero) Cabang Samarinda, baik itu berupa sampel batubara ataupun berupa cangkang kelapa sawit.

3.2 Jadwal Penelitian

Aktifitas penelitian meliputi persiapan, uji pendahuluan, penelitian, dan penyelesaian. Jadwal penelitian dapat di lihat pada table 3.1.
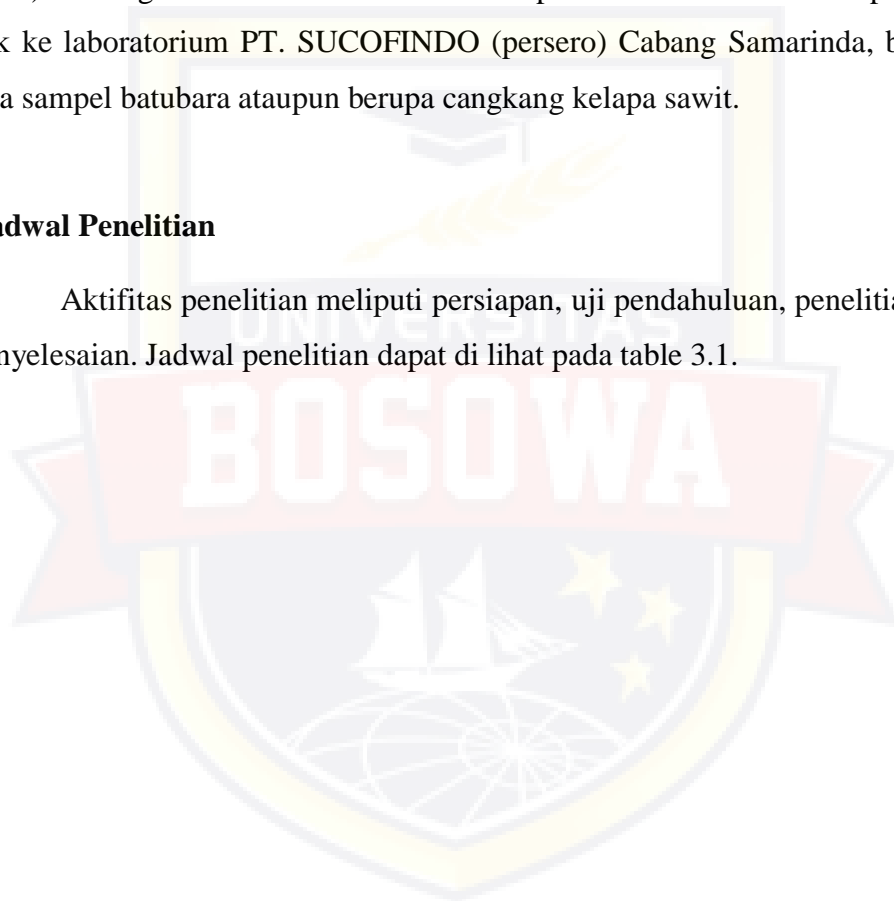
Tabel 3.1. Jadwal Penelitian Karakteristik Batubara Blending Cangkang Kelapa Sawit

No	Kegiatan	Bulan											
		Februari				Maret				April			
		1	2	3	4	1	2	3	4	1	2	3	4
1.	**Persiapan**												
	a. Studi Literatur	■	■										
	b. Penyusunan Proposal		■	■									
	c. Seminar Proposal				■								
2.	**Penelitian Utama**												
	a. Persiapan Alat dan Bahan					■							
	b. Persiapan sampel						■						
	c. Preparasi Sampel							■					
	d. Penentuan nilai Abu							■	■				
	e. Penentuan nilai kalori								■				
	f. Penentuan korelasi								■				
	g. Pembuatan grafik								■				
	h. Pengolahan data								■	■			
3.	**Penyelesaian**												
	a. Pembuatan laporan										■		
	b. Seminar Hasil (Skripsi)											■	

BLENDING DAN ANALISA LABORATORIUM FLOW CHART

Batubara
Bahan Baku

Cangkang Sawit
Bahan Baku

Parameter Analisa :
> Total Moisture standard acuan ASTM D3173 - 2011
> Ash Standard acuan D3174 - 2012
> Total Sulphur standard acuan D4239 - 2012
> Gross Calorific Value standard acuan ASTM 5865-2013

Parameter Analisa :
> Total Moisture standard acuan ASTM D3173 - 2011
> Ash Standard acuan D3174 - 2012
> Total Sulphur standard acuan D4239 - 2012
> Gross Calorific Value standard acuan ASTM 5865-2013

Blending
Proses Batubara blending cangkang kelapa
sawit dengan komposisi :
- batubara 75% dengan cangkang 25 %
- batubara 50 % dengan cangkang 50 %
- batubara 25 % dengan cangkang 75 %

Parameter Analisa :
> Total Moisture standard acuan ASTM D3173 - 2011
> Ash Standard acuan D3174 - 2012
> Total Sulphur standard acuan D4239 - 2012
> Gross Calorific Value standard acuan ASTM 5865-2013

**Produk hasil
Blendingan siap
di gunakan**

Gambar 3.1 : Flow Chart Blendingan Batubara dengan Cangkang Kelapa Sawit.
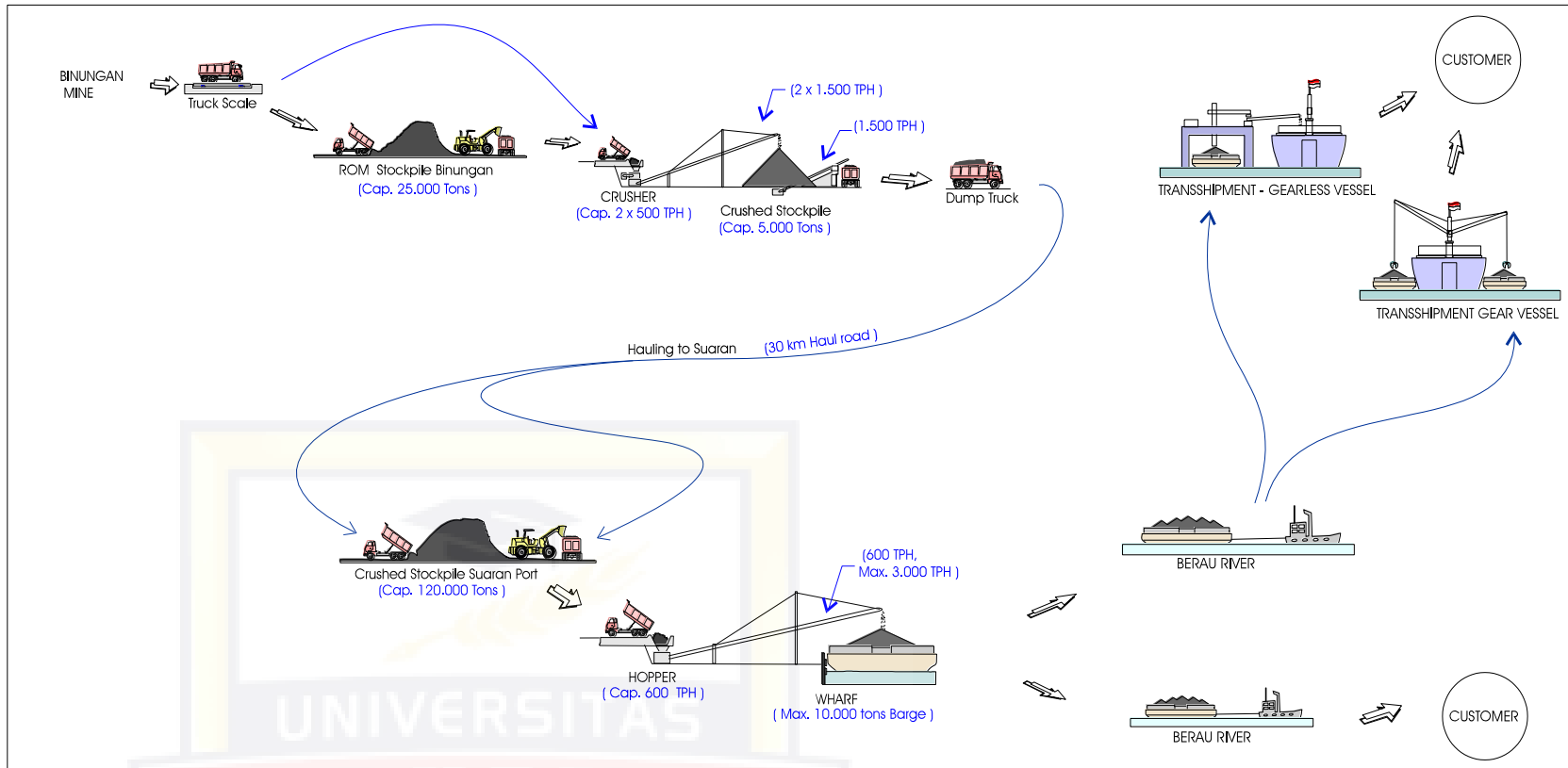
Gambar 3.2. Proses Penanganan dan Pengolahan Batubara Blending Cangkang Kelapa Sawit.

3.3 Rancangan Penelitian.

Karakteristik dari pada batubara blending cangkang kelapa sawit akan di bedakan dalam beberapa variable sebagai berikut :

3.3.1 Moisture in the Analysis Sample Batubara & Cangkang Kelapa Sawit (Kandungan Air Lembab)

3.3.1.1 Standard Acuan

ASTM D3173 - 2011

3.3.1.2 Ruang Lingkup

Metode ini untuk menentukan kadar air lembap dalam batubara & Cangkang kelapa sawit yang akan dianalisa dan metode yang digunakan adalah *ASTM*.

3.3.1.3 Prinsip

Contoh yang telah diketahui massanya dikeringkan dalam oven yang telah dikalibrasi pada suhu 105 - 110 °C. Kadar air lembab dalam contoh dihitung dari massa yang hilang setelah pemanasan.

3.3.1.4 Peralatan

 ➢ Neraca analitik.
 ➢ Desikator.
 ➢ Oven
 ➢ Petridish
 ➢ Stopwatch
 ➢ Tray logam.

3.3.1.5 Bahan

 ➢ Silikagel (Aluminum silika)
 ➢ Gas oksigen.
 ➢ Gas nitrogen.

3.3.1.6 Perlakuan Contoh

Contoh batubara & cangkang kelapa sawit dengan diameter 0.212 mm disimpan dalam ruangan, yang terkontrol suhu dan tekanannya dan bertempat di ruangan timbang.

3.3.1.7 Cara Pelaksanaan

- Naikkan suhu oven hingga suhu terkalibrasi, yaitu : 105 - 110 °C
- Set kecepatan aliran gas (udara kering) hingga 400 cm ^{3}/menit.
- Timbang dan catat massa dari petridish kosong dan tutupnya sebagai M1.
- Timbang contoh 1.0000 ± 0.0010 gr ke dalam petridish.
- Catat massa petridiks, tutupnya dan contoh sebagai M2.
- Buka penutup petridish, letakkan diatas pan logam kemudian masukkan kedalam oven dan dikeringkan selama 1 (satu) jam.
- Ambil petridish dan penutupnya, tutup petridis dengan penutupnya, kemudian dinginkan dalam desikator selama 15 menit.
- Timbang dan catat massa dari petridis, penutupnya dan contoh kering sebagai M3.
- Parameter diatas dikerjakan duplo, dengan waktu yang sama untuk setiap contoh.
- Contoh standar inhouse dianalisa untuk setiap batch pengujian contoh, sebagai verifikasi data.
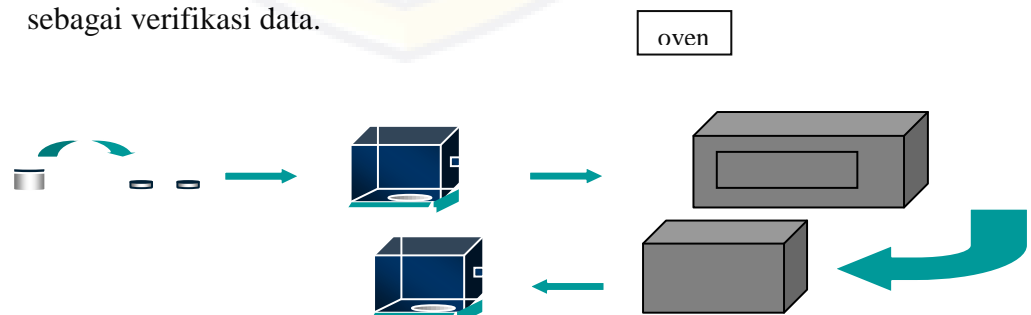
Gambar 3.3: Skema Kerja Pengujian Kandungan Air Lembab

3.3.1.8 Analisis Hasil

Perhitungan kandungan air lembab dalam contoh adalah sebagai berikut:

M % ad = **Error! Reference source not found.**

Hasil yang dilaporkan adalah nilai rata-rata hasil analisa duplo.

M1 = Massa petridish kosong

M2 = Massa petridish + simple sebelum pemanasan

M3 = Massa petridish + simple setelah pemanasan

3.3.2 Ash Content (Kandungan Abu)

3.3.2.1 Standard Acuan

ASTM D3174 – 2012

3.3.2.2 Ruang Lingkup

Metode ini untuk menentukan kandungan abu dari contoh batubara & Cangkang kelapa sawit dengan menggunakan metode standar ASTM.

3.3.2.3 Prinsip

Contoh yang telah diketahui massa-nya dipanaskan dalam *Furnace* mulai suhu ruang hingga suhu 500°C selama 1 (satu) jam, kemudian pemanasan dilanjutkan hingga 750°C selama 1 jam dan dibiarkan pada suhu tersebut selama 90 menit, kandungan abu dihitung dari massa residu.

3.3.2.4 Peralatan

- ➢ Neraca analitik.
- ➢ Cawan porselin.
- ➢ Furnace.
- ➢ Gegep.
- ➢ Kuas.

- ➢ Nampan logam.
- ➢ Spatula.

3.3.2.5 Perlakuan Contoh

Contoh batubara & cangkang kelapa sawit dengan diameter 0.212 mm disimpan dalam ruangan yang terkontrol suhu dan tekanannya dan bertempat di ruangan timbang.

3.3.2.6 Cara Pelaksanaan

- ➢ Timbang dan catat massa dari cawan kosong sebagai M1.
- ➢ Timbang contoh 1.0000 ± 0.0010 gr ke dalam cawan.
- ➢ Catat massa cawan dan contoh sebagai M2.
- ➢ Cawan dimasukkan ke dalam *Furnace* mula-mula pada suhu kamar, menggunakan gegep.
- ➢ Suhu dinaikkan sampai 500 ^{0}C dan dibiarkan selama 1 jam kemudian suhu dinaikkan lagi sampai 750^{0}C dan dibiarkan selama 1,5 jam.
- ➢ Buka sedikit pintu *Furnace* untuk mendinginkan, hingga$\pm$ 300 ^{o} C.
- ➢ Cawan dikeluarkan dari *Furnace* menggunakan gegep dan ditaruh di atas nampan logam kemudian didinginkan sampai suhu kamar.
- ➢ Timbang cawan dan abu sebagai M3.
- ➢ Bersihkan cawan dan timbang ulang sebagai M4.
- ➢ Sample dikerjakan secara duplikat (duplo).
- ➢ Contoh standar *inhouse* dianalisa untuk setiap *batch* contoh, sebagai verifikasi data.
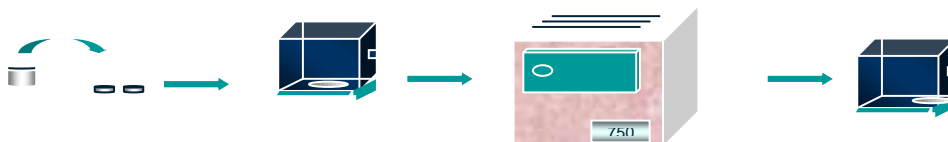
Gambar 3.4: Skema Kerja Pengujian Kandungan Abu

3.3.2.7 Analisis Hasil

Perhitungan kandungan abu (*Ash Content*) adalah sebagai berikut:

Ash % ad = **Error! Reference source not found.**

Hasil yang dilaporkan adalah nilai rata-rata hasil analisa duplo.

M1 = Massa cawan kosong sebelum pemanasan

M2 = Massa cawan + sample sebelum pemanasan

M3 = Massa cawan + abu setelah pemanasan

M4 = Massa cawan kosong setelah pemanasan

3.3.3   Total Sulfur (*TS metode Spektrofotometer Infra Merah*)

3.3.3.1 Standar Acuan

ASTM D4239 – 2012

3.3.3.2 Ruang Lingkup

Metode ini digunakan untuk menentukan kandungan Total Sulfur   dalam sampel menggunakan alat penganalisa sulphur yang bernama LECO.

3.3.3.3 Dasar Prinsip

Sampel yang telah diketahui massanya dibakar dengan adanya oksigen pada suhu 1350°C, yang akan mengubah sulphur menjadi *sulphur oksida*. *Magnesium perchlorate* digunakan sebagai perangkap untuk "*menangkap*" air dan partikel-partikel lain. Aliran gas dilewatkan melalui sel infra merah. Jumlah dari sulphur dalam sampel dihitung dari perbandingan terhadap pengukuran sel sampel dengan pengukuran sel standar yang telah disertifikasi.

3.3.3.4 Peralatan

- ➢ LECO tipe S.144 DR.
- ➢ Cawan perahu.

➢ Neraca analitik.

3.3.3.5 Bahan.

➢ Gas oksigen.

➢ Hablur $Mg(ClO_4)_2$.

➢ Glass woll

3.3.3.6 Cara Pelaksanaan

➢ Tempatkan cawan kosong di atas neraca analitik. Tunggu beberapa detik untuk peneraan otomatis terhadap neraca analitik.

➢ Timbang 0,2000 gram sampel kedalam cawan perahu, jika beratnya telah stabil tekan tombol enter

➢ Letakkan cawan didepan pintu tabung pembakar lalu tekan tombol ANALIZE dan tunggu pesan dari LOAD TANUR. Gunakan tongkat pendorong logam untuk mendorong cawan berisi sampel ke dalam tabung pembakar hingga cawan tersebut menyentuh dinding belakang dari tanur.

➢ Tunggu hingga analisa berjalan sempurna selama 3 menit.

➢ Pindahkan cawan dari tabung pembakar, menggunakan kait dari tongkat pendorong dan biarkan mendingin pada suhu laboratorium.

➢ Lakukan analisa ASCRM *(Australian Standard Certifed Reference Materials)* dan in house sebagai data verifikasi.

➢ Catat analias pada worksheet dan hitung rata-rata perbandingkan sampel terhadap ASCRM.

3.3.3.7 Analisis Hasil

Perhitungan kadar total sulfur adalah sebagai berikut :

% S   =   **Error! Reference source not found.** $\times$ % SO_2
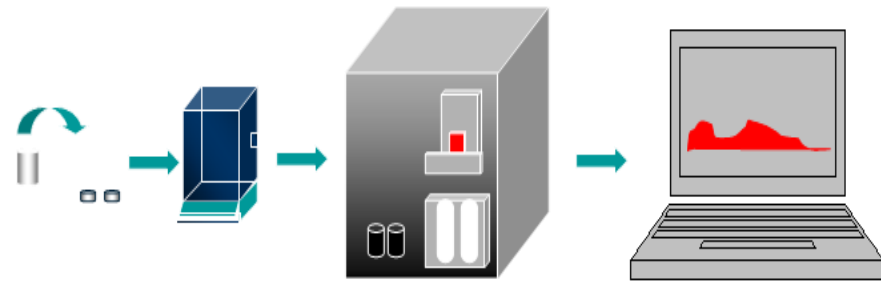
Gambar 3.5 : Skema Kerja Pengujian Total Sulfur

3.3.4 Calorivic Value (Nilai Kalori)

3.3.4.1 Standard Acuan

ASTM D 5865 – 2013

3.3.4.2 Ruang Lingkup

Metode ini adalah untuk menentukan nilai kalori dari contoh batubara & cangkang kelapa sawit menggunakan *Bomb Calorimeter Parr 6200*

3.3.4.3 Prinsip

Contoh yang telah diketahui massanya, dibakar dalam bomb calorimeter pada kondisi standar. Nilai kalori kasar dihitung dari naiknya suhu air di dalam *vessel* kalorimeter dan kapasitas panas rata-rata dari sistem.

3.3.4.4 Peralatan

- ➢ Neraca analitik
- ➢ Bomb calorimeter.
- ➢ Krusibel bomb calorimeter
- ➢ Kawat stainless steel *(fuse)*.

3.3.4.5 Bahan.

- ➢ Gas oksigen
- ➢ Sodium carbonate. (Na_2CO_3).

➢ Indikator merah methyl.

➢ Aquadest.

3.3.4.6 Perlakuan Contoh

Contoh batubara & cangkang kelapa sawit dengan diameter 0.212 mm disimpan dalam ruangan yang terkontrol suhu dan tekanannya dan bertempat di ruangan timbang.

3.3.4.7 Cara Pelaksanaan

➢ Cek kondisi alat, tekanan gas, regulator, volume air pendingin dan aliran listrik.

➢ Nyalakan dengan menekan tombol hitam yang ada dibelakang alat keposisi atas untuk mengaktifkan alat, pompa, pemanas dan laju air.

➢ Buka aliran gas oksigen dengan cara memutar *pulp* hitam kekiri

➢ Tunggu selama ± 20 menit untuk menstabilkan alat.

➢ Timbang *benzoic acid* atau *IHS* dan sampel seberat ± 1.0000 gram ke dalam krusibel.

➢ Tempatkan krusibel pada penyangga *electrode* dan atur kawat pemantik tersentuh/kontak dengan sample.

➢ Satukan *combustion chamber* dengan *bomb cap* dengan cara memutar *bomb cap* ke kanan sampai kencang, dipastikan *combustion chamber* dan *bomb cap* sesuai dengan pasangannya.

➢ Isi gas pada *vessel* dengan oksigen hingga tekanan maksimum 30 atm (tekan tombol FILL)

➢ Masukkan *vessel* ke dalam *bomb bucket* dan isi dengan 2 liter aquadest dari *pipet tank*

➢ Masukkan elekroda pada *terminal nut* dan pastikan kedua elektroda tersebut terkoneksi dengan *terminal nut*.

➢ Tutup *bomb bucket lid* dan pastikan tertutup rapat

➢ Tekan [START] kemudian pilih ID bomb dan dimasukkan berat sampel

- Tunggu sampai proses analisa selesai dan dicatat hasil analisa
- *Bombbucket* yang berisi *vessel* dikeluarkan dari *bomb jacket*
- Keluarkan *vessel* dari *bomb bucket*.
- Buang gas CO2 dengan cara memutar *knop* yang berada di *bombcap*
- Cuci bagian dalam *bomb* dengan air, ditampung air pencuci kedalam labu erlenmeyer. Dibersihkan semua kawat yang tidak terbakar dari elektroda dan dicuci kepala *bomb* dengan air dan ditampung air cucian kedalam labu erlenmeyer yang sama dengan di atas.
- Titrasi air cucian dengan larutan standar Na_2CO_3 menggunakan indikator *methyl merah* hingga mencapai titik akhir berwarna *orange–merah*. Dicatat volume penitar.

3.3.4.8 Analisis Hasil

- Tekan tombol REPORT dan dimasukkan nomor contoh.
- Masukkan volume penitar
- Masukkan nilai total sulphur (TS %ad).
- Laporan akhir dicetak sebagai nilai kalori akhir. Diperiksa bahwa semua detail telah benar dan catat di worksheet.

  Perhitungan secara manual :

  Hc **Error! Reference source not found.**

  e2 **Error! Reference source not found.**$_2SO_4$

  Fk S = **Error! Reference source not found.**

  keterangan : Hc = Gross heat of combustion

  W = Energi equivalen (EE)

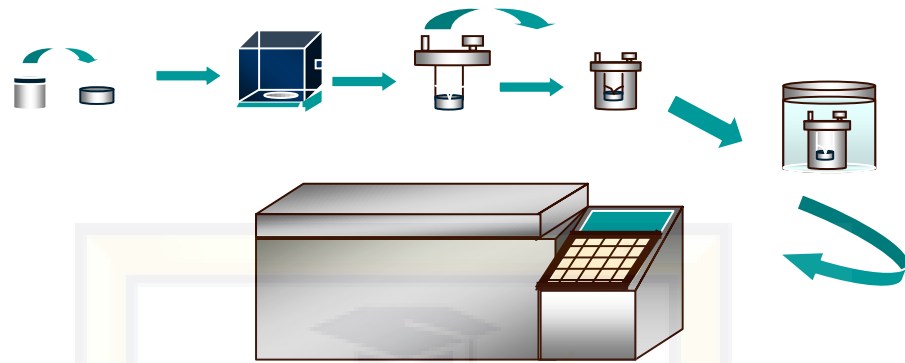 T   = Temperature rise

  e1 = Nilai Acid

  e2 = Nilai sulfur sebenarnya

  e3 = Nilai fuse wire

  S multiplier = 0.6238

  Heat formation of H_2SO_4 = 36.1

Gambar 3.6: Skema Kerja Pengujian Nilai Kalori Batubara

BAB IV

HASIL PENELITIAN DAN PEMBAHASAN

4.1 Hasil Penelitian

Penelitian ini bertujuan untuk mengetahui pengaruh antara batubara dengan cangkang kelapa sawit, apabila ke dua variable material ini di blending. Pada penelitian ini karakteristik dari proses blendingan batubara - cangkang kelapa sawit dapat memberikan manfaat yang kemudian menjadi bahan baku yang berguna dan menguntungkan.. Bahan baku yang akan kita gunakan terlebih dahulu harus di ketahui analisanya agar supaya memudahkan natinya untuk proses pencampuran (blendingan). Parameternya biasa di sebut dengan proksimat analysis seperti kadar air (%), kadar abu (%), kadar sulfur (%)dan kalori (kalori/gr). Berikut data bahan baku batubara dan cangkang kelapa sawit dapat di lihat pada tabel 4.1

Tabel 4.1 Data Bahan Baku Batubara dan Cangkang Kelapa Sawit.

Bahan baku	Kadar air (%)	Kadar abu (%)	Total sulfur (%)	Kalori (kalori/gr)
Batubara	14,30	5,10	0,66	5508
Cangkang kelapa sawit	8,19	5,97	0,04	4475

Dalam proses ini kita lakukan blendingan dengan 3 variasi yaitu :

1. 25% batubara di blending dengan 75% cangkang kelapa sawit.

2. 50% batubara di blending dengan 50% cangkang kelapa sawit.

3. 75% batubara di blending dengan 25% cangkang kelapa sawit.

Hasil dari proses tersebut dianalisa kadar proksimatnya seperti kadar air (%), kadar abu (%), kadar sulfur (%)dan kalori (kalori/gr). Data karakteristik batubara blending cangkang kelapa sawit dapat di lihat pada tabel 4.2

Tabel 4.2 : Data Karakteristik Batubara Blending Cangkang Kelapa Sawit

Blendingan	Kadar air (%)	Kadar abu (%)	Total sulfur (%)	Kalori (kalori/gr)
Batubara 25% dengan Cangkang 75%	9,72	5,71	0,18	4701
Batubara 50% dengan Cangkang 50%	11,28	5,45	0,33	4977
Batubara 75% dengan Cangkang 25%	12,97	5,23	0,52	5199

4.2 Pembahasan

4.2.1 Pengaruh Kadar Air

Kandungan air dalam sample dapat didefinisikan sebagai persentase berat yang hilang jika sample blendingan (batubara dan cangkang kelapa sawit) dipanaskan pada kondisi temperatur standar yakni pada suhu 105°C.

Kandungan air yang tinggi dalam sample blendingan (batubara dan cangkang kelapa sawit) dapat menyebabkan penurunan mutu blendingan sebab:

➢ Menurunkan nilai kalor sample blendingan (batubara dan cangkang kelapa sawit).

➢ Menurunkan titik nyala.

➢ Memperlambat proses pembakaran.

Untuk kadar air sampel blendingan (batubara dan cangkang kelapa sawit), berikut ini grafiknya, pada gambar 4.1
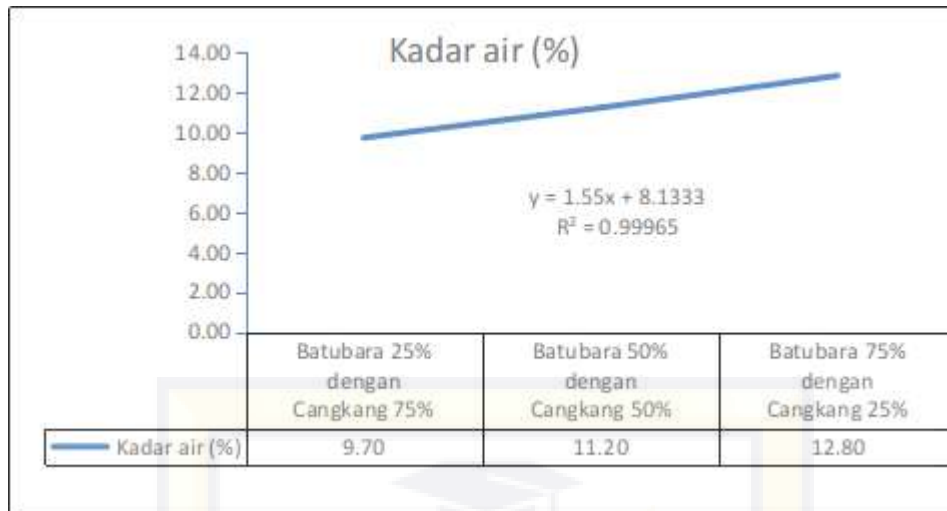
Gambar 4.1 Grafik Hasil Analisa Proksimat Kadar Air Pada Batubara Blending Cangkang Kelapa Sawit
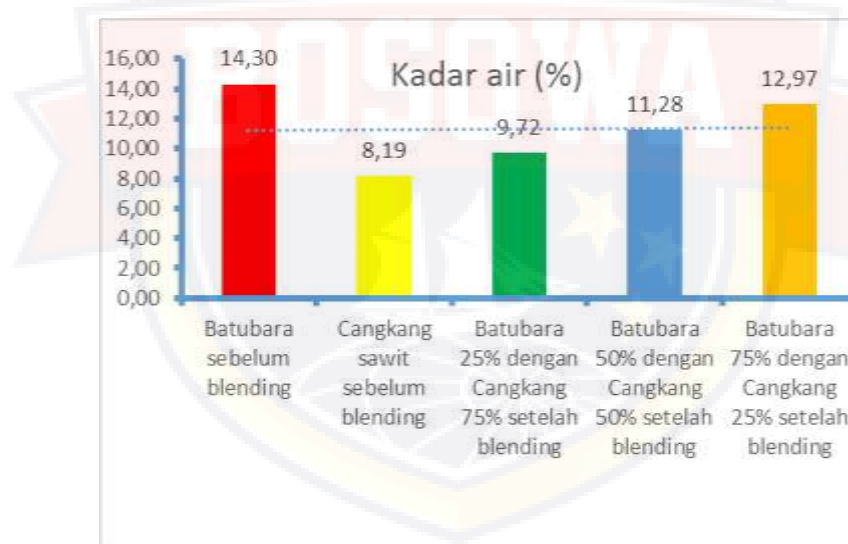
Gambar 4.2 Grafik Kadar Air Pada Batubara dan Cangkang Kelapa Sawit Sebelum dan Sesudah Blendingan

4.2.2 Pengaruh Kadar Abu

Di dalam analisis abu didefinisikan sebagai sisa pembakaran yang tinggal jika dipijarkan material pembentuk abu yang terbentuk dalam sampel terdiri dari dua macam yaitu *inherent* dan e*xtraneous* (tidak

berhubungan dengan materi pembentuk batubara dan cangkang kelapa sawit).

Kadar abu dari sampel penting diketahui sebab:

➤ Kadar abu memberikan indikasi dasar terhadap kekotoran sampel sehingga dapat dipakai sebagai dasar untuk perencanaan kelayakan pembakaran/tanur.

➤ Kadar abu mencerminkan banyaknya mineral dalam sampel dan secara tidak langsung mencerminkan jumlah nilai kalor dari sampel. Bila kadar abu tinggi maka nilai kalor rendah.

Untuk kadar abu sampel blendingan (batubara dan cangkang kelapa sawit), berikut ini grafiknya, pada gambar 4.3
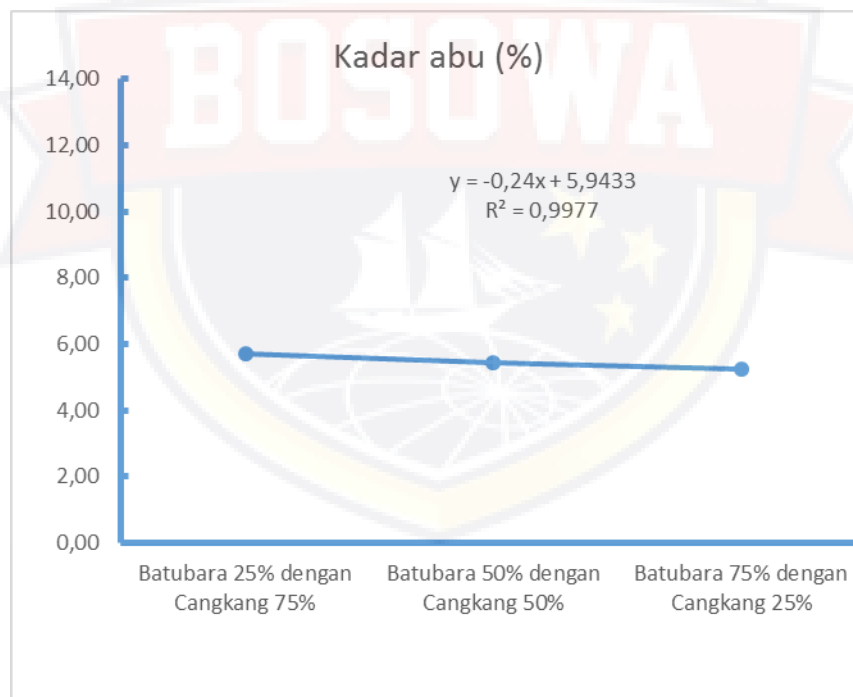
Gambar 4.3 Grafik Hasil Analisa Proksimat Kadar Abu Pada Batubara Blending Cangkang Kelapa Sawit
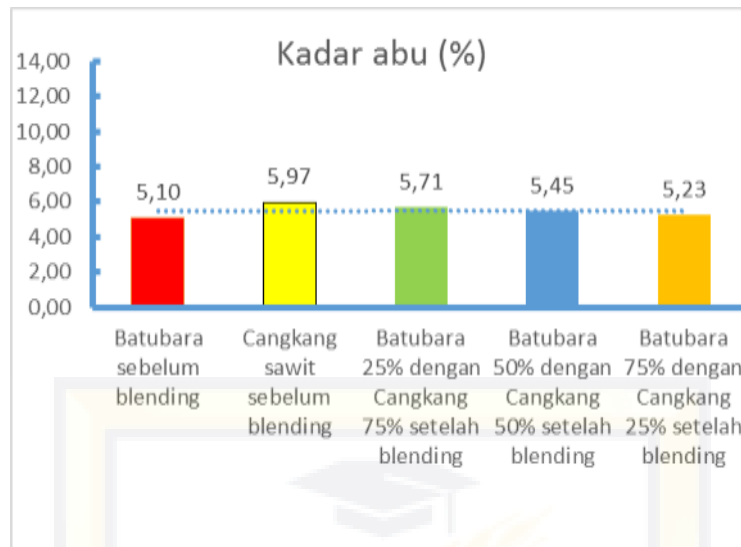
Gambar 4.4 Grafik Kadar Abu Pada Batubara dan Cangkang Kelapa Sawit Sebelum dan Sesudah Blendingan

4.2.3 Pengaruh Kadar Sulfur

Sulfur sangat mempengaruhi kualitas dari suatu produk material yang akan di gunakan sebagai bahan bakar untuk boiler, di karenakan dapat menimbulkan pencemaran udara yang signifikan terhadap lingkungan sekitarnya, sampel yang mengandung sulfur tinggi menimbulkan bau belerang yang menyengat. Semakin tinggi nilai sulfur dari suatu sampel semakin jelek kualitasnya.

Untuk kadar sulfur sampel blendingan (batubara dan cangkang kelapa sawit), berikut ini grafiknya, pada gambar 4.5

Gambar 4.5 Grafik Hasil Analisa Kadar Sulfur Pada Batubara Blending

Cangkang Kelapa Sawit
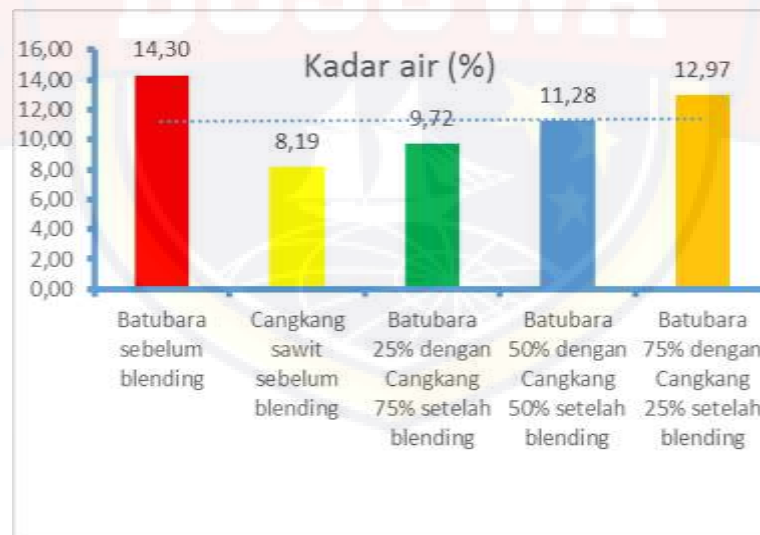
Gambar 4.6 Grafik Kadar Sulfur Pada Batubara dan Cangkang Kelapa

Sawit Sebelum dan Sesudah Blendingan

4.2.4 Pengaruh Kadar Kalori

Nilai kalori adalah jumlah panas yang dihasilkan oleh suatu bahan bakar yang dibakar pada kondisi yang terkendali. *Gross calorific value* adalah jumlah unit panas yang dikeluarkan per unit bahan yang dibakar dengan oksigen di bawah kondisi standar. Nilai kalori biasanya dinyatakan dalam satuan cal/g. Nilai kalori dari sampel ditetapkan dengan cara membakar sampel dalam lingkungan berisi gas oksigen dengan tekanan 30 atm, panas yang dilepaskan oleh pembakaran setimbang dengan nilai kalori sampel.

Untuk kadar abu sampel blendingan (batubara dan cangkang kelapa sawit), berikut ini grafiknya, pada gambar 4.7
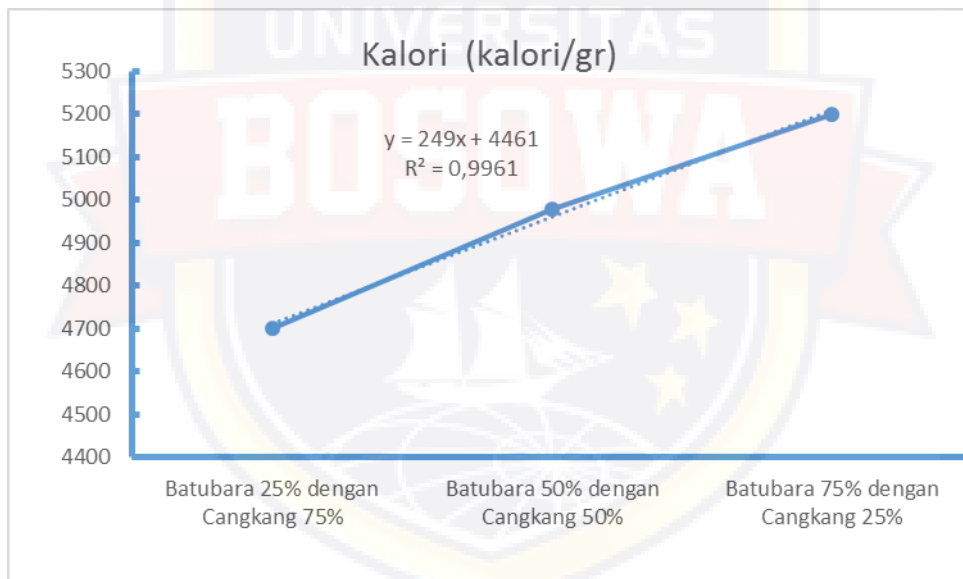
Gambar 4.7 Grafik Hasil Analisa Kadar Kalori Pada Batubara Blending Cangkang Kelapa Sawit
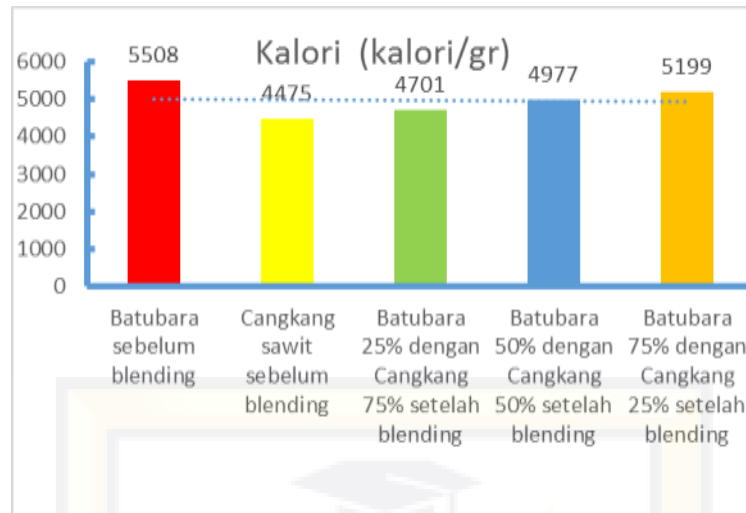
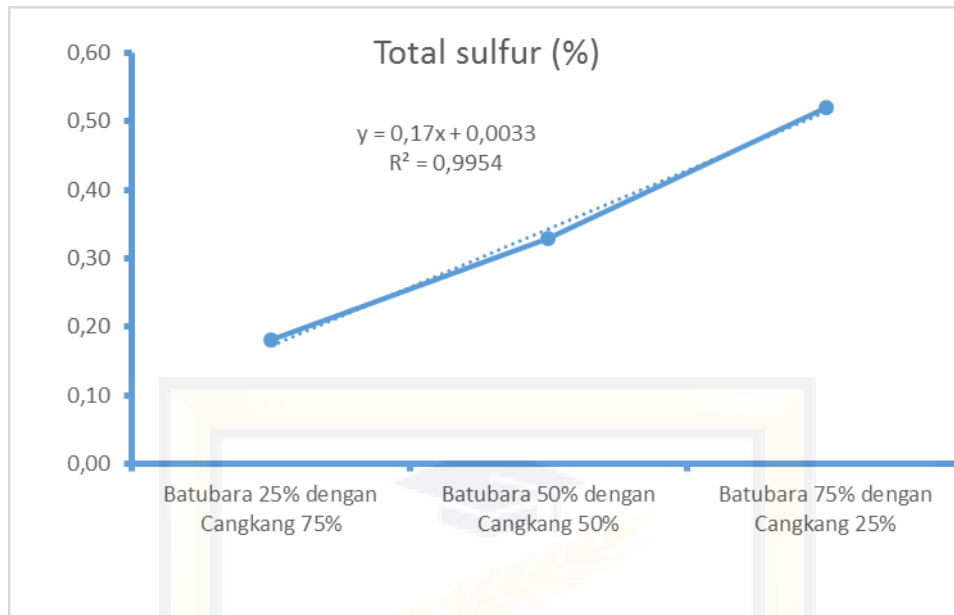**Gambar 4.8 Grafik Kadar Kalori Pada Batubara dan Cangkang Kelapa Sawit Sebelum dan Sesudah Blendingan**
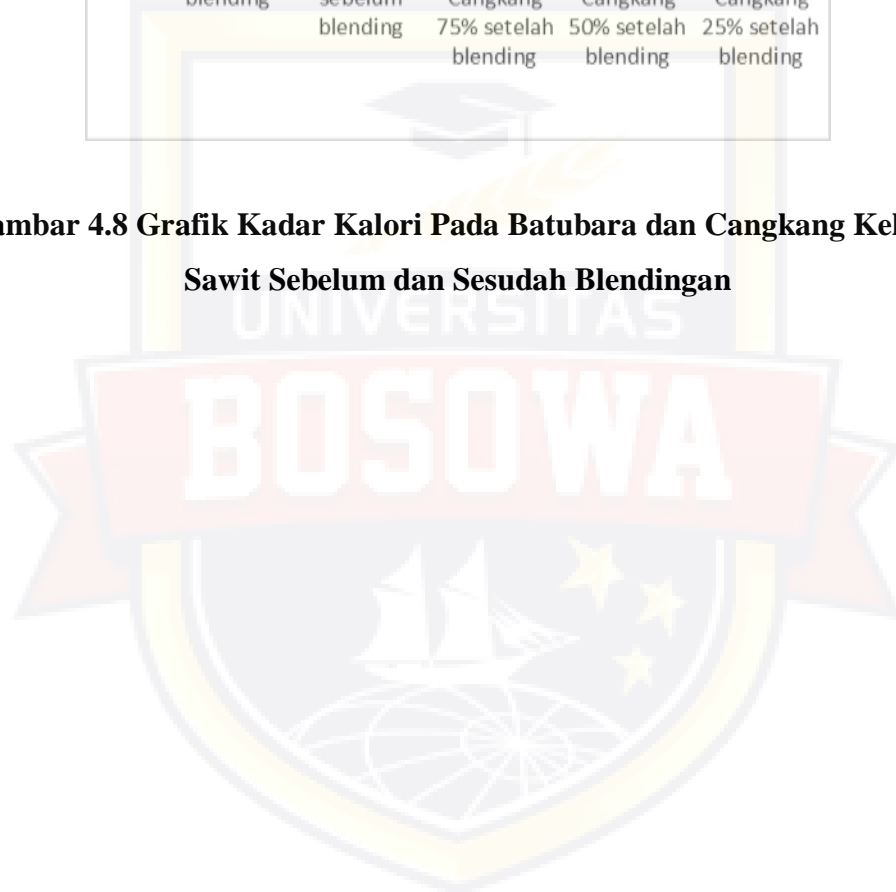
BAB V

KESIMPULAN DAN SARAN

5.1 Kesimpulan

Berdasarkan penelitian yang telah dilakukan, dapat di simpulkan bahwa :

1. Mengubah kondisi optimun rasio yang di timbulkan apabila batubara di blending dengan cangkang kelapa sawit, yang memberikan nilai sulfur signifikan yang ramah lingkungan dari penurunannya mulai 0,52 % ke 0,33 % sampai 0,18 %.

2. Kualitas batubara meningkat dengan sulfur semakin rendah, ramah lingkungan dan cangkang kelapa sawit sebagai limbah menjadi maksimal penggunaannya.

5.2 Saran

1. Perlu dilakukan penelitian lanjutan dengan komposisi blendingan yang berbeda dari batubara dengan cangkang kelapa sawit.

2. Proses analisa harus sesegera mungkin di lakukan untuk mengurangi kesalahan analisa yang terjadi.

DAFTAR PUSTAKA

Agustina. 2006. Land Aplication Sebagai Alternative 3R pada Industri Kelapa Sawit. Kementrian Lingkungan Hidup Indonesia, Jakarta. Hal 45 – 78.

Anonim.*Hand Book* (Instruksi Kerja SBU Jasa Laboratorium).
Standar, Chris. 2003. Coal Quality dan Quality Manajement.

Badan Pusat Statistik. (2015). *Kalimantan Timur Dalam Angka 2015*. Badan Pusat Statistik Kalimantan Timur Ditjen PPHP, Departemen Pertanian. 2006. Pedoman Pengelolaan Limbah Industri Kelapa Sawit. Jakarta. 130 hal.

Fauzi, Y. Yustina EW. Iman S. dan Rudi Hartono. 2014. Kelapa Sawit : Budidaya, Pemanfaatan Hasil dan Limbah, Analisis Usaha dan Pemasaran. Penebar Swadaya. Jakarta. 212 hal.

Hermanto, H. 2013. Pengolahan Limbah Sawit. https://www.wordpress.com. Diakses 15 Juni 2015

Kementerian, L. I. 2015. PROPER. www.menlh.go.id. Diakses 12 Agustus 2015

Kurniawan, D. 2014. Pedoman Pengelolaan Limbah Industri Kelapa Sawit. http://www.academia.edu. Diakses 15 Juni 2015

Loebis, B dan P, L. Tobing. 1992. Potensi Pemanfaatan Limbah Pabrik Kelapa Sawit. Buletin Perkebunan. Pusat Penelitian Kelapa Sawit. Medan. 279

hal.

Mahida. 1984. Limbah Kelapa Sawit. Yogyakarta. 145 hal.
Mulia, A.(2007). *Pemanfaatan Tandan Kosong Dan Cangkang Kelapa Sawit Sebagai Briket Arang* (Tesis). Sumatera Utara: Sekolah Pasca Sarjana, Universitas Medan. 28 Oktober 2015.

Naibaho, P, 2003. Teknologi Pengolahan Kelapa Sawit, Medan : Pusat Penelitian Kelapa Sawit. Medan. 165 hal.

Napi, 2015. Berbagai Pemanfaatan Limbah Kelapa Sawit. http://www.piterest.com/gubuktani.Diakses4Agustus2015

Nurhakim, Y.I. 2014. Perkebunan Kelapa Sawit Cepat Panen. Infra Pustaka. Jakarta.

Pahan, I, 2012, Manajemen Agribisnis dari Hulu hingga Hilir. Penebar Swadaya. Jakarta. 412 hal.

Pardamean, M. 2011. Panduan Lengkap Pengelolaan Kebun dan Pabrik Sawit. Agromedia Pustaka. Jakarta.

Rusmery, T. 2009. Korelasi Antara Biological Oxygen Demand (Bod) Limbah Cair Pabrik Kelapa Sawit Terhadap pH, Total Suspended Solid (Tss), Alkaliniti dan Minyak/ Lemak. Medan. 167 hal.

Said, G. 1996. Penanganan dan Pemanfaatan Limbah Kelapa Sawit. Ungarun Trubus Agriwidya. 89 hal.

Santika. 1987. Metode Penelitian Air. Usaha Nasional. Surabaya. 138 hal.

Sari, H.M. (2015). *Pengaruh Kecepatan Udara Dan Waktu Proses Pirolisis Cangkang Kelapa Sawit Dengan Prinsip Oksidasi Parsial Terhadap Komposisi Produk Cair*(Tugas Akhir). Samarinda: Program Studi Petro Dan Oleo Kimia.Politeknik Negeri Samarinda.

Sasongko, Setia B. 1990. Beberapa Parameter Kimia Sebagai Analisis Air. Edisi keempat. Semarang. 128 hal.

Setyamidjaja D. (2006). *Kelapa Sawit*. 09 Desember 2015. Yogyakarta:Kanisius.

Standard Operating Procedures. 2005. PT. Incasi Raya Jamika Raya. Jambi. 324 hal. Wikipedia. 2013. Limbah industri kelapa sawit.

Yunita Rachma (2016). *Pengaruh Waktu Aktivasi Kimia Terhadap Arang Aktif Cangkang Kelapa Sawit Hasil Pirolisis Oksidasi Parsial* (Tugas Akhir). Samarinda: Program Studi Petro Dan Oleo Kimia. Politeknik Negeri Samarinda.

Zunipar A.F.R. (2015). *Pembuatan Arang Aktif Melalui Proses Pirolisis Dengan Prinsip Oksidasi Parsial Dan Aktivasi Secara Kimia* (Tugas Akhir). Samarinda: Program Studi Petro Dan Oleo Kimia. Politeknik Negeri Samarinda

LAMPIRAN

1. Data Hasil Penelitian.
2. Surat persetujuan Penelitian.
3. Foto-foto penelitian.
4. SOP Asli ASTM method.
5. Lembar Data Keselamatan Bahan.
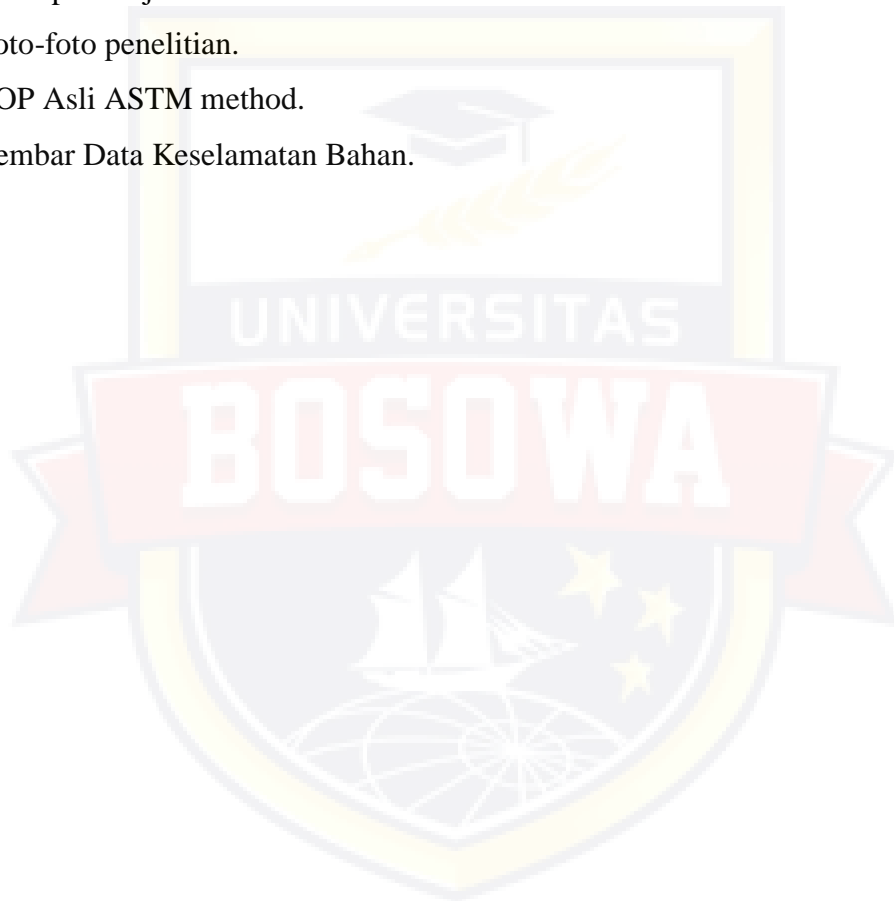
Data Hasil Penelitian

DETERMINATION OF
MOISTURE IN THE ANALYSIS SAMPEL

SUCOFINDO

REFERENCE NO.	: Penelitian UNIBOS	BALANCE ID	:
OVEN ID	: 05	CORECTION BALANCE	:
TIME START – FINISH	: 10.00 – 11.00	STANDARD METHOD	: - ISO 11722-2013
			- ASTM D3173-11

DATE		4/5/17					
SAMPLE ID		CK	CK	P.B	PB		
DISH NO.		15	16	17	18		
M1	gram	14.8312	14.7864	15.6244	15.4328		
M2	gram	15.8317	15.7963	16.6248	16.4230		
M3	gram	15.7497	15.7144	16.4810	16.2890		
% M ad		8.20	8.19	14.29	14.31		
AVERAGE		8.19		14.30			
ANALYZED BY		Elm		Elm.			
VERIFIED BY							

DATE		4/5/17					
SAMPLE ID		25PB + 75 CK	25PB + 75 CK	50PB + 50 CK	50PB + 50CK	75PB + 25CK	75PB + 25CK
DISH NO.		19	20	21	22	23	24
M1	gram	16.1340	15.2144	16.7210	15.4871	15.6622	16.3954
M2	gram	17.1342	16.2148	16.7245	15.4323	15.6625	16.3455
M3	gram	17.6372	16.1194	16.6085	15.3197	15.5329	16.2157
% M ad		9.70	9.74	11.30	11.26	12.96	12.98
AVERAGE		9.72		11.28		12.97	
ANALYZED BY		Elm		Elm		Elm.	
VERIFIED BY							

M1 = Mass of dish
M2 = Mass of dish + sample (before heating)
M3 = Mass of dish + sampel (after heating)

% M ad = [(M2 – M3)/(M2 – M1)] x 100 %

DETERMINATION OF ASH CONTENT

SUCOFINDO

REFERENCE NO.	: Penelitian onnissos
DATE	: 4/5/17
BALANCE ID	: 02
FURNACE ID	: 01
STANDARD METHOD	: ISO 1171 : 2010, ASTM D3174-12
FURNACE INDICATED TEMP	:

SAMPLE ID.		CK		BB			
ANALYST		Slm		Slm			
DISH NO.		01	02	03	04		
M1	g	16.2741	14.4125	15.2243	15.1024		
M2	g	17.2737	15.4127	16.2247	16.1030		
M2 – M1	g	0.9996	1.0002	1.0004	1.0006		
M3	g	16.2836	14.4723	15.3122	15.2104		
M4	g	16.2741	14.4125	15.3636	15.2612		
M3 – M4	g	0.0595	0.0598	0.0512	0.0508		
% ASH		5.95	5.98	5.12	5.08		
AVERAGE		5.97		5.10			

SAMPLE ID.		25 BB + CK 75		50 RB + CK 50		75 BB + 25 CK	
ANALYST		Slm		Slm		Slm	
DISH NO.		05	06	07	08	09	10
M1	g	15.2206	14.4201	16.1120	15.8445	16.2323	15.5621
M2	g	16.2210	15.4206	17.1128	16.8447	17.2330	16.5624
M2 – M1	g	1.0004	1.0005	1.0008	1.0002	1.0007	1.0003
M3	g	15.2779	14.4779	16.1668	15.8996	16.2850	15.6149
M4	g	15.2209	14.4207	16.1125	15.8449	16.2328	15.5625
M3 – M4	g	0.0570	0.0572	0.0543	0.0547	0.0522	0.0524
% ASH		5.70	5.72	5.43	5.47	5.22	5.24
AVERAGE		5.71		5.45		5.23	

M1   : Mass of crucible (Before Heating)
M2   : Mass of crucible + sample (Before Heating)
M3   : Mass of crucible + sample (After Heating)
M4   : Mass of crucible (After Heating)
M2 – M1   : Mass of sample
M2 – M3   : Mass of sample loss on heating

$$\% \text{ ASH CONTENT} = [\,(M3 - M4)\,] / [\,(M2 - M1)\,] \times 100\,\%$$

| Analyzed by: Slm | Verified by: |
| Date : 4/5/17 | Date : |

DETERMINATION OF
SULFUR BY IR SPECTROMETRY

SUCOFINDO

REFERENCE NO.   :   Penelitian Onipos

STANDARD METHOD   :   ISO 19579: 2006

ASTM D4239 - 14

DATE		04/05/17					
BALANCE ID		AB 04					
FURNACE ID		TS 07					
SAMPLE ID		BB 25 + CS 75		BB 50 + CS 50		BB 75 + CS 25	
MASS OF SAMPLE	g	0,2009	0.2008	0.2003	0.2006	0.2001	0.2000
SAMPLE CALL ID		77235	77236	77237	77238	77239	77240
TOTAL SULPHUR % ad		0.12442	0.17220	0.31685	0.38855	1.48660	0.00650
AVERAGE		0.17		0.32		0.50	
CORRECTION		0.32/0.31 = 1.03		1.03		1.03	
RESULTS	% ad	0.18		0.32		0.52	

DATE		04/05/17					
BALANCE ID		AB 04		AB 04			
FURNACE ID		TS 01		TS 07			
SAMPLE ID		BB		CK			
MASS OF SAMPLE	g	0.2004	0.2006	0.2003	0.2005		
SAMPLE CALL ID		77241	77242	77243	77244		
TOTAL SULPHUR % ad		0.64002	0.64124	0.04021	0.04100		
AVERAGE		0.64		0.04			
CORRECTION		1.03		1.03			
RESULTS	% ad	0.64		0.04			

Analyzed by: flm	Verified by:
Date : 4/5/17	Date :

FOR/COAL – OPS/029   Rev : 00   Tanggal berlaku: 13/03/15   Hal 1 dari 1 hal

DETERMINATION OF GROSS CALORIFIC VALUE

SUCOFINDO

REFERENCE NO.	: Penelitian Orisa	CALORIMETER ID	: 07
DATE	: 4/5/17	REFERENCE METHOD	: - ISO 1928 : 2009
			- ASTM D5865 -13

SAMPLE ID.	BB 25 + CK 75		BB 50 + CK 50		BB 75 + CK 25	
BOMB NO.	I	2	1	2	1	2
CALL ID	1695	1696	1697	1698	1699	1900
SAMPLE MASS (g)	1.0001	1.0003	1.0001	1.0005	1.0009	1.0006
Preliminary Result	4730.10	4721.07	4993.24	4949.58	5235.08	5241.40
Spike Mass (g)	-	-	-	-	-	-
Fuse Cal	50	50	50	50	50	50
Acid ml	13	13	12	12	15	15
Total Sulphur %	0.18	0.18	0.33	0.33	0.53	0.53
FINAL RESULT	4702	4700	4976	4978	5148	5200
AVERAGE	4701		4977		5199	
ANALYZED BY	Slin		Slin		Slin	
VERIFIED BY						

SAMPLE ID.	BB		CK			
BOMB NO.	1	2	1	2		
CALL ID	1701	1702	1703	1704		
SAMPLE MASS (g)	1.0002	1.0003	1.0001	1.0004		
Preliminary Result	5513.97	5524.01	4992.11	4436.10		
Spike Mass (g)	-	-	-	-		
Fuse Cal	50	50	50	50		
Acid ml	12	12	13	13		
Total Sulphur %	0.66	0.66	0.04	0.04		
FINAL RESULT	5507	5509	4174	4476		
AVERAGE	5508		4475			
ANALYZED BY	Slin		RS			
VERIFIED BY						

Surat Persetujuan Penelitian

Ref . 031/SMR-III/HC-SCI/2017

Tanggal, 31 Maret 2017

Kepada Yth,
Kaprodi Teknik Kimia
Universitas Bosowa
di-
    Makassar

Dengan Hormat,

Perihal : **Permohonan PKL**

Menindak lanjuti Surat Saudara No. 022/SKL/FT/TK/UNIBOS/III/2017 Perihal Permohonan Ijin Penelitian atas nama :

1. **Salman**

Bersama ini di informasikan kami dapat menyetujui permohonan tersebut sebagaimana waktu yang ditentukan.

Bagi Mahasiswa yang melaksanakan PKL harus taat kepada aturan perusahaan yang berlaku dan mempunyai disiplin dan dedikasi yang tinggi sehingga dalam pembinaan akan menghasilkan SDM yang baik dan diakui.

Demikian atas perhatian dari bapak kami ucapkan terima kasih.

Hormat Kami,

Maksum
Human Capital

Foto-foto Penelitian

LAMPIRAN

DOKUMENTASI KEGIATAN

Photo : 1.

Keterangan : Analisa Sampel penelitian untuk parameter kandungan air lembab.

Photo : 2.

Keterangan : Analisa Sampel penelitian untuk parameter kandungan Abu.

Photo : 3.

Keterangan : Analisa Sampel penelitian untuk parameter kandungan Total Sulfur.
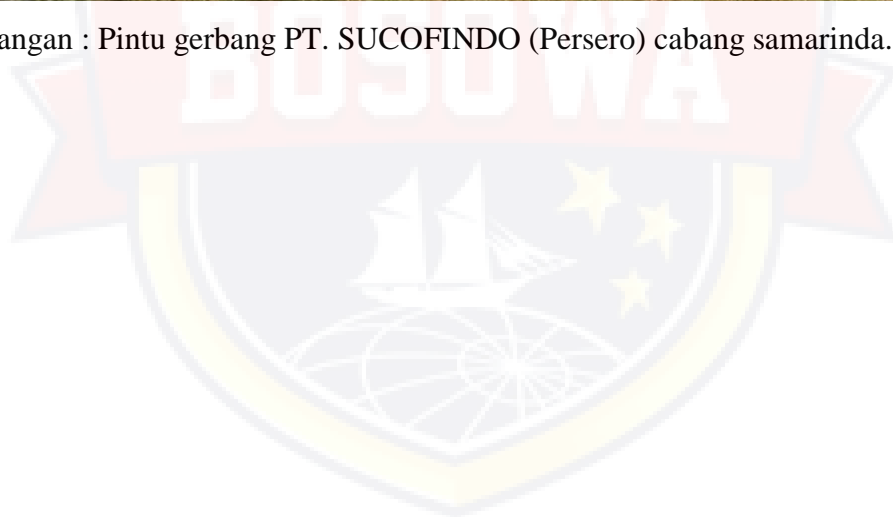
Photo : 4.

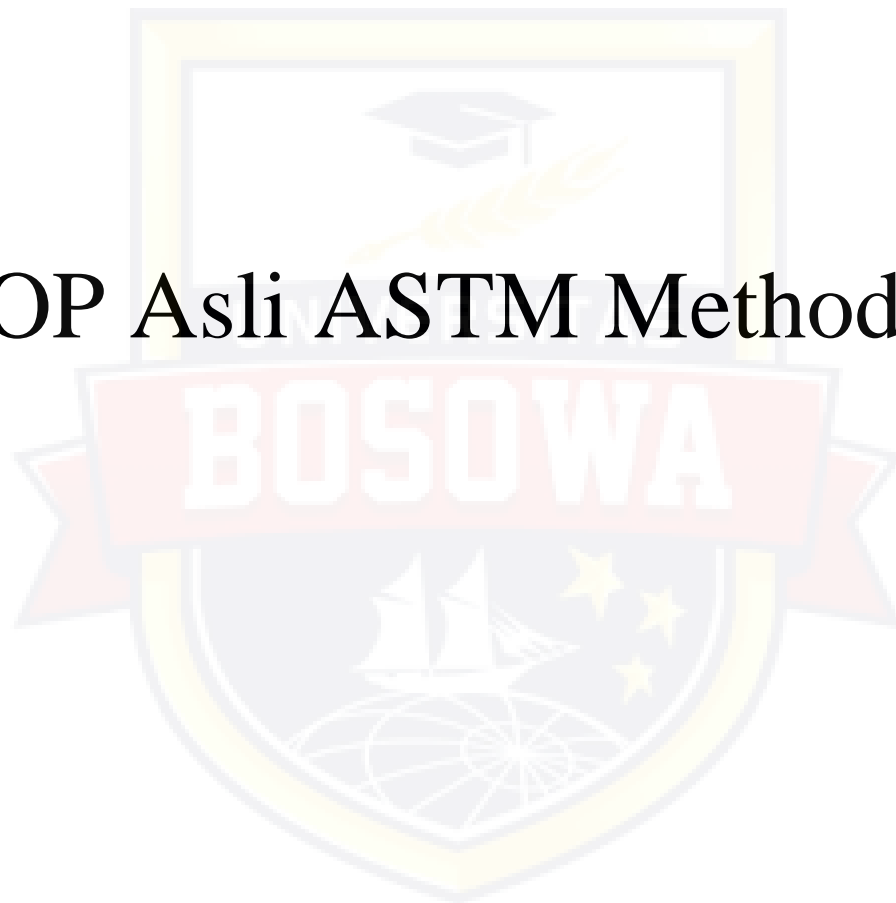Keterangan : Analisa Sampel penelitian untuk parameter kandungan Nilai Kalori.

Photo : 5

Keterangan : Pintu gerbang PT. SUCOFINDO (Persero) cabang samarinda.

SOP Asli ASTM Method

Standard Test Method for
Moisture in the Analysis Sample of Coal and Coke[1]

This standard is issued under the fixed designation D3173; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of moisture in the analysis sample of coal or coke. It is used for calculating other analytical results to a dry basis. When used in conjunction with the air drying loss as determined in accordance with Method D2013 or Practice D346, each analytical result can be calculated to an as-received basis:

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D2013 Practice for Preparing Coal Samples for Analysis
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
D3302 Test Method for Total Moisture in Coal

3. Summary of Test Method

3.1 Moisture is determined by establishing the loss in weight of the sample when heated under rigidly controlled conditions of temperature, time and atmosphere, sample weight, and equipment specifications.

4. Significance and Use

4.1 Moisture as determined by this test method is used for calculating other analytical results to a moisture free basis using procedures in Practice D3180. Moisture percent determined by this test method may be used in conjunction with the air dry moisture loss determined in Method D2013 and Test

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Method D3302 to determine total moisture in coal. Total moisture is used for calculating other analytical results to "as received" basis using Practice D3180. Moisture, ash, volatile matter, and fixed carbon percents constitute the proximate analysis of coal and coke.

5. Analysis Sample

5.1 The analysis sample is that sample which has been pulverized to pass 250-μm (No. 60) sieve as prepared in accordance with Practice D346 or Method D2013.

6. Apparatus

6.1 *Drying Oven*, for coal samples.

6.1.1 For determining the moisture of coal, the oven shall be so constructed as to have a uniform temperature in all parts, have a minimum of air space, and be capable of temperature regulation between the limits of 104 and 110°C. It may be of the form shown in Fig. 1. Provision shall be made for renewing the preheated air in the oven at the rate of two to four times a minute, with the air dried as defined in 7.1.

6.1.2 In the oven shown in Fig. 1, the door should contain a hole of approximately 3.2 mm (⅛ in.) in diameter near the bottom to permit a free flow of air through the oven space.

6.2 *Drying Oven*, for coke samples. For determining the moisture of coke, an ordinary drying oven with openings for natural air circulation and capable of temperature regulation between limits of 104 and 110°C may be used.

6.3 *Capsules*, with covers. A convenient form, which allows the ash determination to be made on the same sample, is a porcelain capsule, 22 mm (⅞ in.) in depth and 44 mm (1¾ in.) in diameter, or a fused silica capsule of similar shape. These capsules shall be used with a well-fitting flat aluminum cover, illustrated in Fig. 2. Platinum crucibles or glass capsules with ground-glass caps may also be used. They should be as shallow as possible, consistent with convenient handling.
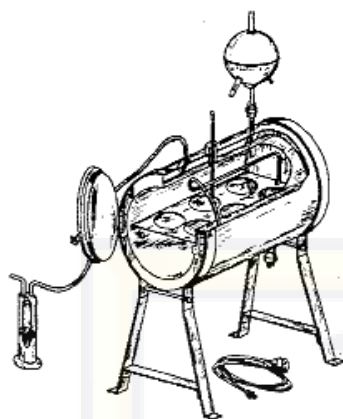
7. Reagents

7.1 *Dry Air*—Air used to purge the drying oven should be dried to a moisture content of 1.9 mg/L or less. (Dew point −10°C or less.) Any desiccant or drying method capable of achieving this degree of dryness is suitable.

7.2 *Desiccants*—Materials suitable for use in the desiccator may be chosen from the following:

TABLE 1 Precision

	Range	Repeatability Limit (r)	Reproducibility Limit (R)
Coal	1.0 – 23.9 %	0.09 + 0.01$\bar{x}$[A]	0.23 + 0.02$\bar{x}$[A]
Coke	0.18 – 1.87%	0.08	0.24

[A]Where $\bar{x}$ is the average of two single test results.

Note 1—Details in U.S. Bureau of Mines Bulletin No. 492, 1951, p 6.
FIG. 1 Moisture Oven

FIG. 2 Capsule for Use in Determining Moisture

7.2.1 *Anhydrous Calcium Sulfate* (0.004 mg/L).
7.2.2 *Silica Gel.*
7.2.3 *Magnesium Perchlorate* (0.0005 mg/L).
7.2.4 *Sulfuric Acid, Concentrated* (0.003 mg/L).
7.2.5 The desiccant must be kept fresh enough to assure that the air in the desiccator is dry as defined in 7.1. Values in parentheses ( ) are literature values for the residual amount of moisture in air at equilibrium with these desiccants. (**Warning**: Sulfuric acid is corrosive and can cause severe damage to eyes, skin, and clothing. Magnesium perchlorate is a strong oxidant and can react violently with organic materials.)

8. Procedure for Sample Passing a 250-µm (No. 60) Sieve

8.1 Heat the empty capsules under the conditions at which the sample is to be dried, place the stopper or cover on the capsule, cool over a desiccant for 15 to 30 min, and weigh. Dip out with a spoon or spatula from the sample bottle approximately 1 g of the sample. Put this quickly into the capsule, close, and weigh at once to the nearest ±0.1 mg.

8.2 An alternative procedure for weighing the sample (more subject to error) is as follows: After transferring an amount of the sample slightly in excess of 1 g, bring to exactly 1 g in weight (±0.5 mg) by quickly removing the excess weight of

the sample with a spatula. The utmost dispatch must be used to minimize the exposure of the sample until the weight is determined.

8.3 After removing the covers, quickly place the capsules in a preheated oven (at 104 to 110°C) through which passes a current of dry air. (The current of dry air is not necessary for coke.) Close the oven at once and heat for 1 h. Open the oven, cover the capsules quickly, cool in a desiccator over desiccant, and weigh as soon as the capsules have reached room temperature.

8.4 Use the percentage of moisture in the sample passing a 250-µm (No. 60) sieve to calculate the results of the other analyses to a dry basis.

9. Calculations

9.1 Calculate the percent moisture in the analysis sample as follows:

$$\text{Moisture in analysis sample, } \% = [(A - B)/A] \times 100 \quad (1)$$

where:
A = grams of sample used and
B = grams of sample after heating.

10. Precision and Bias[3]

10.1 *Precision*—The precision of this method for the determination of moisture in the analysis sample of coal and coke is shown in Table 1 and Table A1.2. The precision characterized by repeatability (S_r, r) and reproducibility (S_R, R) is described in Table A1.1 in Annex A1.

10.1.1 *Repeatability Limit (r)*—The value below which the absolute difference between two test results calculated to separate consecutive test determinations, carried out on the same sample in the same laboratory, by the same operator, using the same apparatus on samples taken at random from a single quantity of homogenous material, may be expected to occur with a probability of approximately 95%.

10.1.2 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results, carried out in different laboratories, using samples taken at random from a single quantity of material that is as nearly homogenous as possible, may be expected to occur with a probability of approximately 95%.

[3] An interlaboratory study, designed consistent with Practice E691, was conducted in 1995. Twelve labs participated. Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1026. In addition, information for coke was calculated per Practice E691 using 48 sets of data for 24 coke samples from a commercially available interlaboratory proficiency test program. The cokes used in this study included furnace coke, foundry coke and coke breeze. Details and supporting information are given in Research Report RR:D05-1030.

10.2 *Bias*—Certified Reference Materials are not available for the determination of bias by this test method.

NOTE 1—One coke sample was analyzed in the interlaboratory study which generated the first row of information in Table 1. The average moisture of the coke sample was 0.36 %. The repeatability limit for this sample is 0.10. The reproducibility limit for this sample is 0.15. The repeatability standard deviation for this sample is 0.036. The reproducibility standard deviation for this sample is 0.053.

ANNEX

(Mandatory Information)

A1. PRECISION STATISTICS

A1.1 The precision of this test method, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the following materials as listed in Table A1.1.

A1.2 *Repeatability Standard Deviation* (S_r)—The standard deviation of test results obtained under repeatability conditions.

A1.3 *Reproducibility Standard Deviation* (S_R)—The standard deviation of test results obtained under reproducibility conditions.

TABLE A1.1 Repeatability (S_r, r) and Reproducibility (S_R, R)
Parameters Used for Calculation of Precision Statement for Coal
Samples

NOTE 1—The R-Squared statistic for both the repeatability (r) and reproducibility (R) indicates that the model as fitted explains 62% of the variability in the two variables.

Material	Average	S_r	S_R	r	R
91-2Mb	0.0683	0.0479	0.0053	0.1341	0.2388
91-1 hvAb	1.5589	0.0569	0.1387	0.1503	0.3864
91-5 hvAb	3.1178	0.0569	0.0977	0.1554	0.2736
89-4 hvCb	5.4426	0.0601	0.1408	0.1682	0.3943
91-4 hvCb	9.6392	0.0636	0.1004	0.1781	0.2811
90-1 subB	11.9	0.1252	0.2193	0.3505	0.614
89-7 subA	12.4556	0.0798	0.1188	0.2234	0.3326
91-5 subA	15.1733	0.0577	0.1737	0.1615	0.4865
89-5 subC	21.3461	0.1949	0.2843	0.5456	0.796

3

TABLE A1.2 Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters Used for Calculation of Precision Statement for Coke Samples

Material	Average	S_r	S_R	r	R
CK980108 - Furnace	0.19	0.04	0.08	0.12	0.22
CK980312 - Furnace	0.30	0.05	0.15	0.13	0.41
CK980716 - Foundry	0.27	0.03	0.10	0.06	0.29
CKReference2 - Furnace	0.16	0.03	0.11	0.09	0.30
CKNIST01 - Foundry	0.57	0.04	0.09	0.13	0.24
CKNIST02 - Furnace	0.30	0.03	0.06	0.09	0.18
CK0303 - Foundry	0.75	0.03	0.06	0.09	0.16
CK9903 - Foundry	0.50	0.04	0.10	0.10	0.29
CK9904 - Furnace	0.65	0.03	0.10	0.09	0.27
CK0001 - Furnace	0.44	0.03	0.07	0.09	0.21
CK0401 - Furnace	0.58	0.03	0.10	0.07	0.30
CK0002 - Breeze	0.46	0.02	0.15	0.04	0.42
CK0302 - Breeze	0.55	0.04	0.05	0.10	0.13
CK0003 - Foundry	0.31	0.02	0.08	0.07	0.24
CK0101 - Furnace	0.19	0.02	0.08	0.04	0.23
CK0504 - Furnace	0.29	0.04	0.08	0.10	0.23
CK0701 - Furnace	0.20	0.02	0.06	0.06	0.24
CK0102 - Furnace	0.33	0.04	0.10	0.10	0.29
CK0301 - Furnace	0.34	0.01	0.08	0.04	0.23
CK0404 - Furnace	0.50	0.02	0.07	0.06	0.21
CK0103 - Furnace	1.04	0.03	0.30	0.07	0.84
CK0304 - Furnace	1.49	0.02	0.10	0.05	0.27
CK0601 - Furnace	1.54	0.03	0.05	0.08	0.14
CK0603 - Furnace	1.87	0.07	0.08	0.18	0.23
CK0104 - Furnace	0.20	0.01	0.08	0.04	0.23
CK0503 - Furnace	0.35	0.02	0.06	0.06	0.17
CK0201 - Furnace	0.51	0.03	0.08	0.08	0.22
CK0501 - Furnace	0.59	0.03	0.13	0.08	0.36
CK0802 - Furnace	0.72	0.02	0.08	0.05	0.23
CK0604 - Furnace	0.77	0.03	0.08	0.08	0.24
CK0202 - Furnace	0.35	0.02	0.13	0.06	0.36
CK0502 - Furnace	0.46	0.02	0.11	0.06	0.32
CK0603 - Furnace	0.53	0.04	0.08	0.10	0.23
CK0203 - Furnace	0.21	0.02	0.08	0.05	0.24
CK0403 - Furnace	0.24	0.02	0.07	0.06	0.20
CK0604 - Furnace	0.23	0.03	0.09	0.07	0.26
CK0801 - Furnace	0.18	0.02	0.05	0.06	0.14
CK0204 - Breeze	0.60	0.04	0.19	0.10	0.54
CK0402 - Breeze	0.72	0.04	0.10	0.12	0.27
CK0602 - Breeze	0.72	0.03	0.14	0.07	0.40
CK0704 - Breeze	0.67	0.02	0.08	0.05	0.22
CK0702 - Furnace	0.64	0.06	0.24	0.18	0.67
CK0902 - Furnace	0.91	0.04	0.18	0.11	0.50
CK0703 - Coke	1.68	0.04	0.11	0.11	0.30
CK0803 - Furnace	0.43	0.02	0.10	0.06	0.28
CK0804 - Furnace	0.44	0.02	0.06	0.06	0.17
CK0901 - Coke	0.27	0.03	0.06	0.09	0.18
CK1001 - Furnace	0.29	0.03	0.11	0.09	0.30

Designation: D3174 – 12

Standard Test Method for
Ash in the Analysis Sample of Coal and Coke from Coal[1]

1. Scope

1.1 This test method covers the determination of the inorganic residue as ash in the analysis sample of coal or coke as prepared in accordance with Practice D2013 or Practice D346 The results obtained can be applied as the ash in the proximate analysis, Practice D3172, and in the ultimate analysis, Practice D3176. For the determination of the constituents in ash, reference is made to Test Methods D3682, D4326 and D6349. Test Method D6357 should be used to prepare ash to be used for trace element analysis. See Terminology D121 for definition of ash.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D121 Terminology of Coal and Coke
D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D388 Classification of Coals by Rank
D1757 Test Method for Sulfate Sulfur in Ash from Coal and Coke (Withdrawn 2009)[3]
D2013 Practice for Preparing Coal Samples for Analysis
D2795 Test Methods for Analysis of Coal and Coke Ash (Withdrawn 2001)[3]

D3172 Practice for Proximate Analysis of Coal and Coke
D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
D3176 Practice for Ultimate Analysis of Coal and Coke
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
D3682 Test Method for Major and Minor Elements in Combustion Residues from Coal Utilization Processes
D4326 Test Method for Major and Minor Elements in Coal and Coke Ash By X-Ray Fluorescence
D5016 Test Method for Total Sulfur in Coal and Coke Combustion Residues Using a High-Temperature Tube Furnace Combustion Method with Infrared Absorption
D6349 Test Method for Determination of Major and Minor Elements in Coal, Coke, and Solid Residues from Combustion of Coal and Coke by Inductively Coupled Plasma—Atomic Emission Spectrometry
D6357 Test Methods for Determination of Trace Elements in Coal, Coke, and Combustion Residues from Coal Utilization Processes by Inductively Coupled Plasma Atomic Emission Spectrometry, Inductively Coupled Plasma Mass Spectrometry, and Graphite Furnace Atomic Absorption
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D121

4. Summary of Test Method

4.1 Ash is determined by weighing the residue remaining after burning the coal or coke under rigidly controlled conditions of sample weight, temperature, time, atmosphere, and equipment specifications

5. Significance and Use

5.1 Ash, as determined by this test method, is the residue remaining after burning the coal and coke. Ash obtained differs in composition from the inorganic constituents present in the original coal. Incineration causes an expulsion of all water, the loss of carbon dioxide from carbonates, the conversion of iron pyrites into ferric oxide, and other chemical reactions. Ash, as

CROSS SECTION OF FURNACE AND PIPES

COAL ASH MUFFLE

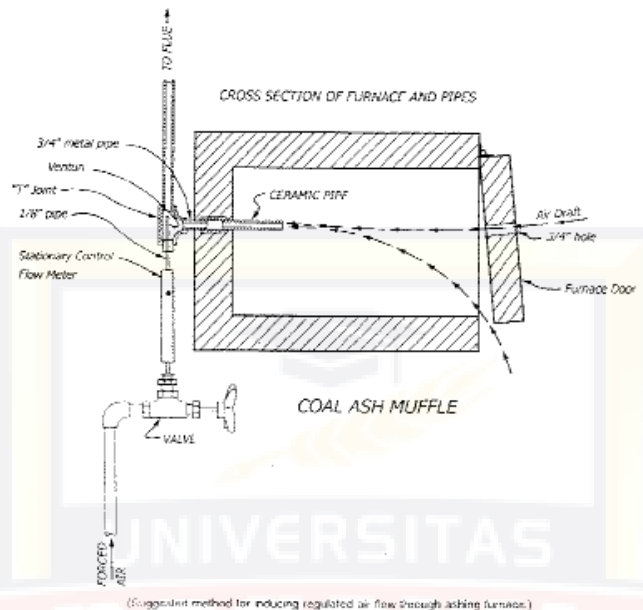(Suggested method for inducing regulated air flow through ashing furnace.)

FIG. 1 Air Aspirator

determined by this test method, will differ in amount from ash produced in furnace operations and other firing systems because incineration conditions influence the chemistry and amount of the ash. References for correcting ash results determined by this test method to a mineral-matter-free basis are listed in Classification D388, Section 9.

6. Apparatus

6.1 *Electric Muffle Furnace for Coal or Coke*—For determination of ash of coal and coke, the furnace shall have an adequate air circulation and be capable of having its temperature regulated at 500°C, 750°C, and 950°C. The furnace shall be equipped with a temperature indicator and means of controlling the temperature within prescribed limits. Means shall be provided for maintaining air flow at a rate of two to four changes per minute (see Fig. 1 and Fig. 2). Combustion gases shall be vented from laboratory. Inlet and outlet ports shall be located and arranged to distribute the air uniformly throughout the furnace area without the possibility of sweeping solid particles from the capsules. The temperature over the entire working area of the furnace floor shall be maintained within the specified temperature limits.

6.2 *Porcelain Capsules*, about 22 mm (⅞ in.) in depth, and 44 mm (1¾ in.) in diameter, or similar shallow dishes or platinum crucibles.

6.3 *Balance*, sensitive to 0.1 mg.

6.4 *Crucible Cover*, aluminum, porcelain, or similar covers.

7. Temperature Calibration

7.1 Place a preignited capsule with 1 g of sand at the center of the working area of the furnace, and by the use of a potentiometer and thermocouple or other suitable temperature measuring device, measure the temperature of the sand in the crucible. The crucible and sand should be at temperature equilibrium with the furnace. There should be two to four air changes per minute moving throughout the furnace (the air flow may be measured by using a wet-test meter or equivalent calibrated at standard conditions for air connected to the ceramic-pipe exhaust). Adjust the furnace temperature until the potentiometer reads 750 ± 10°C and then adjust or read the temperature on the indicating pyrometer. Use this reading as the proper setting for controlling the furnace.

8. Procedure

8.1 The sample shall be the material pulverized to pass No. 60 (250-mm) sieve in accordance with Practice D2013 or Practice D346.

8.2 Transfer approximately 1 g (weighed to the nearest 0.1 mg) of the thoroughly mixed sample to a weighed capsule and

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CROSS SECTION OF FURNACE AND PIPES
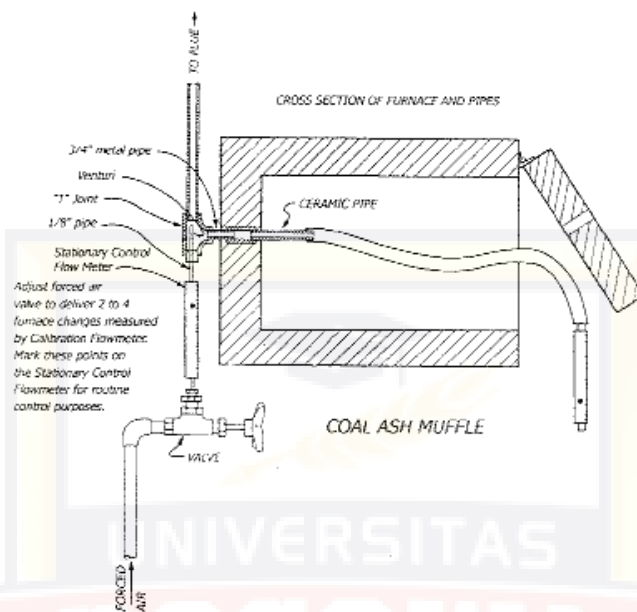
Calibration Flowmeter with Tubing – Ambient Air - For calibration use only, adjust forced air valve to deliver two to four furnace volume changes per minute (at standard temperature-pressure conditions.)

Note 1—Flowmeters are usually calibrated for one atmosphere at 70°F (760-mm Hg at 21.1°C).

(Suggested layout for calibration.)

FIG. 2 Air Aspirator

cover quickly. An alternative way is to use the dried coal from the moisture determination in Test Method D3173. After removing the covers, place the capsule containing the sample in a cold furnace and heat gradually at such a rate that the temperature reaches 500 ± 10°C at the end of 1 h.

8.3 For coals continue heating the sample until the temperature rises from 500 + 10°C to 750 ± 15°C at the end of 1 hour. For cokes continue heating the sample until the temperature rises from 500 ± 10 C to 950 ± 20°C at the end of 1 hour. Continue to heat at the final temperature (750°C or 950°C) for an additional 2 h. Remove the capsule from the muffle furnace, place the cover on the capsule, cool under conditions to minimize moisture pickup, and weigh.

8.4 The two-stage ashing procedure allows pyritic sulfur to be oxidized and expelled before most metal carbonates are decomposed. An ample supply of air in the muffle furnace, "two to four volume changes per minute," must be assured at all times to ensure complete oxidation of the pyritic sulfur and to remove the SO₂ formed.

8.5 While the 4-h incineration interval described is sufficient with most coals to reach a condition of complete burn off, certain cokes and nonreactive coals may require additional time. If unburned carbon particles are observed, or if duplicate results are suspect, the samples should be returned to the furnace for sufficient time to reach a constant weight (±0.001 g). The 4-h time limit may be reduced if the sample reaches a constant weight at 750°C to 950°C in less than 4 h.

8.6 Some samples may be encountered that contain a high amount of carbonates (calcite) or pyrites or both. In such cases, sulfur retained as sulfates may be both unduly high and nonuniform between duplicate samples. In such cases, sulfate sulfur in the ash can be determined in accordance with Test Method D5016 and the value properly corrected. If such is done, the ash value should be reported and designated both as-determined and corrected.

TABLE 1 Determination of Ash in the Analysis Sample of Coal and Coke

	Range	Repeatability Limit, r	Reproducibility Limit, R
Coal	2.08 to 17.86 %	0.22	0.32
Coke	5.73 to 11.75 %	0.10	0.20

TABLE 2 Range and Limits for Repeatability and Reproducability for the Detrmination of Ash in Coal and Coke

Coal	Range	Repeatability Limit, r	Reproducibility Limit, R
Bituminous	5.0 to 15.0 %	0.30 %	0.48 %
Subbituminous-Lignite	4.5 to 30.0 %	0.33 %	0.47 %

9. Calculation

9.1 Calculate the ash percent in the analysis sample as follows:

$$\text{Ash in analysis sample, \%} = [(A - B)/C] \times 100 \qquad (1)$$

where:

A = weight of capsule, cover, and ash residue, g,
B = weight of empty capsule and cover, g, and
C = weight of analysis sample used, g.

10. Report

10.1 For reporting analyses to other than as-determined basis, refer to Practice D3180.

11. Precision and Bias

11.1 *Precision*

11.1.1 *250 μm (No. 60) Samples*—The precision of this test method for the determination of ash in the analysis sample of coal and coke is shown in Table 1. The precision characterized by repeatability (S_r, r) and reproducibility (S_R, R) is described in Table A1.1 and Table A1.2 in Annex A1.

11.1.1.1 *Repeatability Limit (r)*—The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) of separate and consecutive test determinations, carried out on the same sample of 250 μm (No. 60) coal and coke in the same laboratory, by the same operator, using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

11.1.1.2 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180), carried out in different laboratories, using samples of 250 μm (No. 60) coal and coke taken at random from a single quantity of material that is as homoge-

neous as possible, may be expected to occur with a probability of approximately 95 %.

11.1.2 *2.36 mm (No. 8) Samples[4]*—The precision of this test method for the determination of ash in the analysis sample of coal and coke is shown in Table 2.

11.1.2.1 *Repeatability Limit (r)*—The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) of separate and consecutive test determinations, carried out on the same sample, using the same riffle, determined on a single test specimen of two separate 2.36 mm (No. 8) test units of coal reduced entirely to 250 μm (No. 60) and prepared from the same bulk sample coal in the same laboratory, by the same operator, using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

11.1.2.2 *Reproducibility Limit (R)*—The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180), carried out in different laboratories, using samples of 2.36 mm (No. 8) coal reduced entirely to 250 μm (No. 60), taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

11.2 *Bias*—Since this is an empirical test method, the degree of absolute bias cannot be determined.

12. Keywords

12.1 ash; coal; coke

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[4] An interlaboratory study, designed consistent with Practice E691, was conducted in 1989. Eight laboratories participated in this study. Supporting data are available from ASTM Headquarters. Request Report RR:D05-1015. In addition, information for coke was calculated per Practice E691 using 48 sets of data for 24 coke samples for a commercially available interlaboratory proficiency test program. The cokes used in this study included furnace coke, foundry coke and coke breeze. Details and supporting information are given in RR:D05-1039.

ANNEX

(Mandatory Information)

A1. PRECISION STATISTICS 250 µm (No. 60) SAMPLES

A1.1 The precision of this test method, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the following materials as listed in Table A1.1 and Table A1.2.

A1.2 *Repeatability Standard Deviation* (S_r)—The standard deviation of test results obtained under repeatability conditions.

A1.3 *Reproducibility Standard Deviation* (S_R)—The standard deviation of test results obtained under reproducibility conditions.

TABLE A1.1 Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters Used for Calculation of Precision Statement for Coal Samples

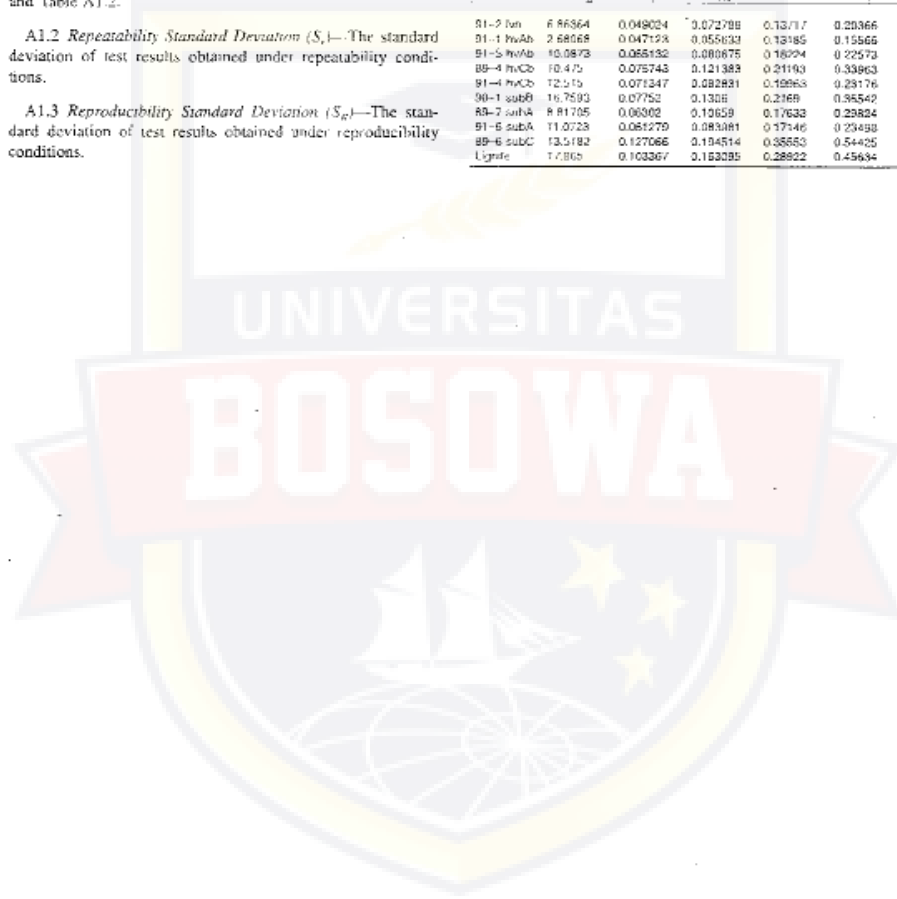| Material	Average	S_r	S_R	r	R
91–2 lvb	6.96364	0.049024	0.072788	0.13717	0.20366
91–1 hvAb	2.68068	0.047123	0.055633	0.13189	0.15566
91–5 hvAb	10.0873	0.065132	0.080675	0.18224	0.22573
89–4 hvCb	10.475	0.075743	0.121383	0.21193	0.33963
91–4 hvCb	72.515	0.071347	0.082831	0.19963	0.23176
90–1 subB	16.7593	0.07752	0.1306	0.2169	0.36542
89–7 subA	9.81705	0.06302	0.10659	0.17633	0.29824
91–6 subC	11.0723	0.061279	0.083081	0.17146	0.23458
89–6 subC	13.5182	0.127066	0.194514	0.35553	0.54425
Lignite	17.865	0.103367	0.163085	0.28922	0.45634

TABLE A1.2 Repeatability (S_r, r) and Reproducibility (S_R, R)
Parameters Used for Calculation of Precision Statement for Coke
Samples

Material	Average	S_r	S_R	r	R
CK980706 - Furnace	6.68	0.03	0.05	0.09	0.15
CK980312 - Furnace	6.74	0.04	0.08	0.11	0.22
CK980716 - Foundry	6.54	0.05	0.08	0.13	0.22
CKReference2 - Furnace	6.08	0.03	0.04	0.08	0.10
CKNIST01 - Foundry	5.73	0.03	0.07	0.10	0.21
CKNIST02 - Furnace	8.03	0.03	0.08	0.09	0.22
CK0303 - Foundry	6.26	0.03	0.06	0.08	0.17
CK9903 - Foundry	6.31	0.04	0.06	0.12	0.18
CK9904 - Furnace	9.22	0.04	0.07	0.10	0.21
CK0001 - Furnace	7.75	0.03	0.07	0.08	0.21
CK0401 - Furnace	7.53	0.04	0.11	0.12	0.30
CK0002 - Breeze	9.05	0.02	0.05	0.07	0.15
CK0302 - Breeze	9.01	0.03	0.11	0.09	0.31
CK0003 - Foundry	6.34	0.04	0.05	0.12	0.15
CK0101 - Furnace	8.05	0.04	0.09	0.13	0.25
CK0504 - Furnace	8.04	0.04	0.05	0.10	0.15
CK0701 - Furnace	8.03	0.04	0.09	0.12	0.25
CK0102 - Furnace	7.45	0.04	0.06	0.11	0.18
CK0301 - Furnace	7.42	0.03	0.04	0.08	0.13
CK0404 - Furnace	7.39	0.02	0.08	0.06	0.24
CK0103 - Furnace	9.28	0.04	0.06	0.12	0.17
CK0304 - Furnace	9.28	0.04	0.05	0.13	0.16
CK0501 - Furnace	9.36	0.03	0.06	0.08	0.17
CK0903 - Furnace	9.31	0.03	0.08	0.08	0.22
CK0104 - Furnace	7.24	0.02	0.06	0.05	0.17
CK0503 - Furnace	7.21	0.02	0.07	0.06	0.19
CK0201 - Furnace	9.10	0.03	0.11	0.07	0.32
CK0501 - Furnace	8.16	0.03	0.05	0.07	0.13
CK0802 - Furnace	8.10	0.05	0.10	0.14	0.28
CK0904 - Furnace	9.09	0.03	0.05	0.09	0.14
CK0202 - Furnace	9.06	0.02	0.04	0.07	0.11
CK0502 - Furnace	8.06	0.03	0.06	0.07	0.16
CK0603 - Furnace	8.02	0.03	0.07	0.07	0.18
CK0203 - Furnace	9.31	0.04	0.14	0.11	0.39
CK0403 - Furnace	8.33	0.04	0.09	0.11	0.24
CK0604 - Furnace	8.27	0.03	0.11	0.09	0.32
CK0801 - Furnace	8.30	0.03	0.11	0.08	0.32
CK0204 - Breeze	7.91	0.04	0.08	0.12	0.22
CK0402 - Breeze	7.92	0.03	0.07	0.09	0.20
CK0602 - Breeze	7.89	0.05	0.09	0.13	0.26
CK0704 - Breeze	7.69	0.04	0.12	0.11	0.33
CK0702 - Furnace	7.56	0.04	0.15	0.10	0.42
CK0902 - Furnace	7.40	0.03	0.06	0.09	0.16
CK0703 - Coke	9.08	0.04	0.11	0.12	0.31
CK0803 - Furnace	11.73	0.03	0.14	0.07	0.39
CK0804 - Furnace	11.75	0.05	0.14	0.14	0.41
CK0901 - Coke	8.04	0.03	0.07	0.09	0.20
CK1001 - Furnace	7.22	0.03	0.06	0.09	0.17

Standard Test Method for
Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion[1]

ε¹ NOTE—Summary of Changes added and Tables 4 and A2.3 corrected editorially in November 2014.
ε² NOTE—Added research report footnotes to Tables A3.7, A3.8, and A3.9 editorially in March 2015.

1. Scope

1.1 This test method covers the determination of sulfur in samples of coal or coke by high-temperature tube furnace combustion.

1.1.1 Two analysis methods are described.

1.2 When automated equipment is used, either method can be classified as an instrumental method.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 All percentages are percent mass fractions unless otherwise noted.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D2013 Practice for Preparing Coal Samples for Analysis
D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
D3176 Practice for Ultimate Analysis of Coal and Coke
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases

D3740 Practice for Establishing the Competence of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke
D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis
E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standard:*[3]
ISO 11722 Solid Mineral Fuels-Hard Coal - Determination of Moisture in the general analysis test sample by drying in nitrogen

3. Summary of Test Method

3.1 *Combustion Method A (1350°C)*—A weighed test portion of sample is burned in a tube furnace at a minimum combustion tube operating temperature of 1350°C in a stream of oxygen. During combustion at temperatures above 1350 °C, the sulfur and sulfur compounds contained in the sample are decomposed and oxidized almost exclusively to gaseous sulfur dioxide, SO₂. Moisture and particulates are removed from the gas by filters. The gas stream is passed through a cell in which sulfur dioxide is measured by an infrared (IR) absorption detector. Sulfur dioxide absorbs IR energy at a precise wavelength within the IR spectrum. Energy is absorbed as the gas passes through the cell body in which the IR energy is being transmitted; thus, at the detector, less energy is received. All other IR energy is eliminated from reaching the detector by a precise wavelength filter. Thus, the absorption of IR energy can be attributed only to sulfur dioxide whose concentration is proportional to the change in energy at the detector. One cell is used for both a reference and a measurement chamber. Total sulfur as sulfur dioxide is detected on a continuous basis.

3.1.1 One procedure for Method A uses coal or coke reference materials to calibrate the sulfur analyzer. A second

procedure for Method A uses a pure substance, BBOT, to calibrate the sulfur analyzer.

3.2 *Combustion Method B (1150°C)*—A weighed test portion of sample is burned in a quartz combustion tube in a stream of oxygen with an equal or excess weight of tungsten trioxide (WO₃). Sulfur is oxidized during the reaction of the sample and WO₃. The tube furnace is operated at a minimum combustion tube operating temperature of 1150°C and tin (Sn) sample boats are utilized. Moisture and particulates are removed from the combustion gas by filters. The gas stream is then passed through a cell in which sulfur dioxide is measured by an infrared (IR) absorption detector. Sulfur dioxide absorbs IR energy at a precise wavelength within the IR spectrum. Energy is absorbed as the gas passes through the cell body in which the IR energy is being transmitted; thus, at the detector, less energy is received. All other IR energy is eliminated from reaching the detector by a precise wavelength filter. Thus, the absorption of IR energy can be attributed only to sulfur dioxide whose concentration is proportional to the change in energy at the detector. One cell is used as both a reference and a measurement chamber. Total sulfur as sulfur dioxide is detected on a continuous basis.

4. Significance and Use

4.1 Sulfur is part of the ultimate analysis of coal and coke.

4.2 Results of the sulfur analysis are used for evaluation of coal preparation and cleaning, evaluation of potential sulfur emissions from coal and coke combustion or conversion processes, and evaluation of coal and coke quality in relation to contract specifications, as well as for scientific purposes.

4.3 The competency of laboratories with respect to use of this standard can be established through reference to Practice D7448.

5. Sample

5.1 Pulverize the sample to pass No. 60 (250-µm) sieve and mix thoroughly in accordance with Practice D2013 or Practice D346.

5.2 Analyze a separate portion of the analysis sample for moisture content in accordance with Test Method D3173, or D7582 or ISO 11722 for calculations to other than the as-determined basis.

5.3 Procedures for calculating as-determined sulfur values obtained from the analysis sample to other bases are described in Practices D3176 and D3180.

6. Apparatus

Combustion Method A (1350°C)

6.1 *Measurement Apparatus*—Equipped to combust the sample as described in 3.1 (See Fig. 1).

6.2 *Tube Furnace*—Capable of heating the hot zone or outer surface of the combustion tube, or both (6.3) to at least 1350°C. It is normally heated electrically using resistance rods, a resistance wire, or molybdenum disilicide elements. Specific dimensions can vary with manufacturer's design.

6.3 *Combustion Tube*—Made of mullite, porcelain, or zircon with provisions for routing the gases produced by combustion through the infrared cell. The tube may have a boat stop made of reticulated ceramics heated to 1350°C that serves to complete the combustion of sulfur containing materials.

6.4 *Sample Combustion Boats*, made of iron-free material and of a convenient size suitable for the dimensions of the combustion tube.
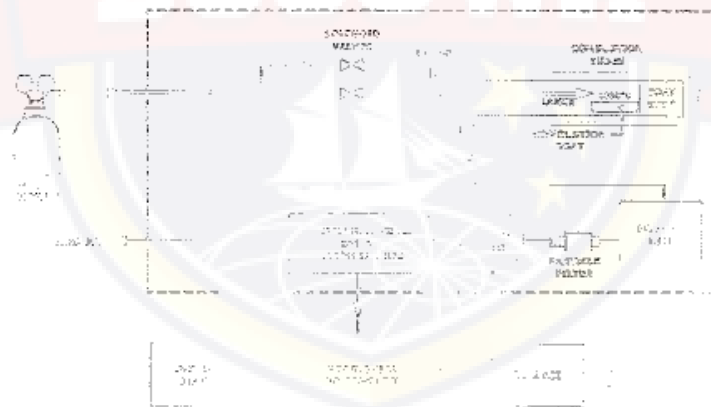

FIG. 1 Apparatus for the Determination of Sulfur by Infrared Detection, Method A

6.5 *Boat Puller*—Where required, a rod of a heat-resistant material with a bent or disk end to insert and remove boats from the combustion tube.

6.6 *Balance*—A stand-alone balance or a balance integrated with the instrument, with a resolution of at least 0.1% relative of the test portion mass.

Combustion Method B (1150°C)

6.7 *Measurement Apparatus*—Equipped to combust the sample as described in 3.2 (See Fig. 2).

6.8 *Tube Furnace*—Capable of heating the hot zone or outer surface of the combustion tube, or both (6.9) to at least 1150°C. It is normally heated electrically using resistance wire. Specific dimensions can vary with manufacturer's design.

6.9 *Combustion Tube*—Made of quartz with provisions for routing the gases produced by combustion through the infrared cell.

6.10 *Sample Combustion Boat*—Made of an iron-free tin material and of a convenient size suitable for the dimensions of the combustion tube.

7. Reagents

7.1 *Purity of Reagents*—Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Available Reagents of the American Chemical Society, where such specifications are available.[4] Other grades can be used, provided it is first ascertained the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Magnesium Perchlorate* (**Warning**—Magnesium perchlorate is a strong oxidizing agent. Do not regenerate the absorbent. Do not allow contact with organic materials or reducing agents.)

7.3 *Oxygen, 99.5 % Pure*—Compressed gas contained in a cylinder equipped with a suitable pressure regulator and a needle valve to control gas flow. (**Warning**—Pure oxygen vigorously accelerates combustion. Verify all regulators, lines, and valves are free of grease and oil.)

7.4 *Reference Materials, Reference Material (RM)*— that are coal(s) or coke(s) prepared by a national metrology body. Other materials that are coal(s) or coke(s) with documented traceability to reference material (CRM) coal(s) or coke(s) prepared by a national metrology body can also be used. Only use material(s) with an assigned value and assigned uncertainty for sulfur. The uncertainty expressed as the confidence interval of the assigned value shall be less than the repeatability specified in the appropriate section on Precision and Bias of this test method.

7.4.1 To minimize problems with instrument calibration or calibration verification mix all reference material before removing the test portion from the container. Do not use the reference material for calibration or calibration verification when less than 2 g remain in the container. The remaining material can be used for instrument conditioning.

7.5 *BBOT* (2,5-di(5-tert-butylbenzoxazol-2-yl)thiophene, $C_{26}H_{26}N_2O_2S$)— A pure substance and certified reference material for sulfur (7.47 % sulfur).

7.6 *Tungsten Oxide (WO_3)*—A combustion promoter and a fluxing agent. (**Warning**—Tungsten Oxide is a strong oxidizing agent.)

8. Procedure

8.1 *Instrument Preparation*—Perform apparatus set up system checks in accordance with manufacturer's instructions.

8.1.1 *Balance Calibration*—Calibrate the instrument balance in accordance with manufacturer's instructions.

8.2 *Calibration of the Infrared Detection System*—If the instrument has been previously calibrated in accordance with the section on instrument calibration, proceed to the Analysis Procedure, otherwise carry out a calibration as specified in the following section.

8.2.1 *Calibration with Coal and Coke Certified Reference Materials*—Select reference materials (7.4), in the range of the samples to be analyzed. Use at least three such reference

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[4] *Reagent Chemicals, American Chemical Society Specifications* , American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

FIG. 2 Apparatus for the Determination of Sulfur by the Infrared Detection, Method B

materials, for each range of sulfur values to be tested. Select one reference material containing at least as much sulfur as the highest level of sulfur expected. Select two additional reference materials, one approximately at the mid-point of the range and one below the lowest level of sulfur expected.

8.2.1.1 Use a mass of material recommended by the apparatus manufacturer to carry out a minimum of three determinations to condition the equipment before calibration. Use a material with a sulfur value near the mid point of the expected calibration range.

8.2.1.2 For each reference coal or coke employed for calibration, use the as-determined sulfur value previously calculated from the certified dry-basis sulfur value and residual moisture determined using either Test Methods D3173 or D7582 or ISO 11722. Use a mass of material and the calibration procedure recommended by the apparatus manufacturer. Weigh to at least the nearest 1 mg and evenly spread the test portion of the reference material into the sample combustion boat (6.4). Position the sample in the hot zone of the furnace until the instrument returns to baseline as indicated according to settings recommended by the manufacturer. If the analysis time exceeds the maximum analysis time recommended by the manufacturer take corrective action as recommended by the manufacturer.

8.2.2 *Calibration with BBOT*—To meet the precision requirements of this method, six calibration points are required for a linear fit and eight calibration points are required for a nonlinear fit. A calibration point consists of a determination on a single test portion of calibration material. Select test portions of the calibrant that have at least as much sulfur as the highest level of sulfur expected, test portions of the calibrant that have as much sulfur as the lowest level of sulfur expected and test portions spread evenly in between the highest and lowest levels of sulfur.

8.2.2.1 The mass of the calibrant needed can be calculated using the following equation: (Note 1).

$$M_C = \frac{(M_T \times S_{AD})}{S_C} \qquad (1)$$

Where

M_C = Mass of calibrant
M_T = Mass normally used for test samples
S_{AD} = Percent sulfur (as-determined) in the test sample
S_C = Percent sulfur in the pure substance calibrant

Note 1—In the interlaboratory study that yielded the data for the precision statement for this method, the mass of BBOT used for calibration ranged from about 15 mg to over 80 mg. Some analyzers may use larger amounts.

8.2.3 *Calibration Verification* Carry out a minimum of three determinations to condition the equipment before calibration verification (see 8.2.1.1). Verify the instrument calibration prior to analyzing test samples, upon completion of all test samples and as needed to meet quality control requirements. Analyze a test portion of reference material(s) (7.4) using the apparatus conditions employed for instrument calibration (8.2.1). Use a mass that does not exceed the maximum mass used for instrument calibration and with a sulfur value within the range of the instrument calibration. If the value determined for each reference material employed for verification is not within the specified uncertainty for the assigned sulfur value repeat the instrument calibration in accordance with 8.2.1. Repeat all samples analyzed since the last successful calibration verification.

8.3 *Combustion Method A (1350°C)*—Set up the apparatus (see 8.1) and verify the calibration (see 8.2.3).

8.3.1 Raise the furnace temperature as recommended by the manufacturer to at least 1350°C. Weigh a mass of the sample to at least the nearest 1 mg and not exceeding the maximum mass of reference material(s) used for calibration. Analyze the test samples using the apparatus conditions employed for calibration (8.2.1).

8.3.2 When the analysis is complete, the instrument indicates the sulfur value.

8.4 *Combustion Method B (1150°C)*—Set up the apparatus (see 8.1) and verify the calibration (see 8.2.3).

8.4.1 Raise the furnace temperature as recommended by the manufacturer to at least 1150°C.

8.4.2 Weigh to the nearest 1 mg and evenly spread into a combustion boat (6.10) a portion of tungsten oxide equal to the target weight, typically 100 mg, of the sample analysis aliquot.

8.4.3 While evenly spreading the sample material into the same combustion boat, weigh to the nearest 1 mg a sample aliquot equal (± 10mg) to the weight of tungsten oxide.

8.4.4 For high-rank (Bituminous and Coke) materials add additional tungsten oxide as required to ensure that its weight is 10 mg (± 5 mg) in excess of the sample aliquot weight measured.

8.4.5 For low-rank (Sub-bituminous and lignite and high ash) test samples or any material of unknown BTU content, add additional tungsten oxide as required to ensure that its weight is at least double (± 10 mg) the sample aliquot weight measured.

8.4.6 For test sample materials, do not exceed the maximum mass of reference material(s) used for calibration. Analyze the test samples using the apparatus conditions employed for calibration.

8.4.7 Follow the manufacturer's recommended combustion sample boat handling procedures to position the sample into the hot zone of the furnace and start the analysis.

8.4.8 When the analysis is complete, the instrument indicates the sulfur value.

9. Calculation to Other Bases

9.1 The percent sulfur value obtained is on an as-determined basis.

9.2 Procedures for converting the as-determined value to other bases are described in Practices D3176 and D3180.

10. Report

10.1 Report the following information:

10.1.1 Mass percent sulfur, as determined, dry, or other bases.

10.1.2 Method used; Results were obtained according to Test Method D4239, (Method A, and calibrants, or Method B).

11. Precision and Bias

11.1 *Precision-250 µm (No. 60) Samples*—The precision of this method for the determination of Sulfur in the 60 mesh sample of coal and coke is shown in Table 1 and Table 2 (Method A)[5] and Table 3 (Method B)[6]. The precision is characterized by repeatability (S_r, r) and reproducibility (S_R, R) and is described in Table A1.1, Table A1.2, Table A1.3 and Table A1.4 in Annex A1.

11.1.1 *Repeatability Limit (r)*—the value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) of separate and consecutive test determinations, carried out on the same sample in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

11.1.2 *Reproducibility Limit (R)*—the value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) carried out in different laboratories using samples taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

11.2 *Bias*—Bias is minimized when reference material(s) are employed to calibrate the instrument.

[5] An interlaboratory study, designed consistent with ASTM Practice E691, was conducted in 1999. Twelve laboratories participated in this study. Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1020. In addition, an interlaboratory study, designed consistent with ASTM Practice E691, was conducted in 2012. Eight laboratories participated in this study. Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1042

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1041

TABLE 1 Method A (Calibration with Coal Reference Materials)—Repeatability and Reproducibility for Sulfur in 250 µm (No.60) Coal

	Range, %	Repeatability Limit (r)	Reproducibility Limit (R)
Coal	0.28–5.61	$0.02 + 0.03x^A$	$0.02 + 0.09x^A$

[A] Where x is the average of two single test results.

TABLE 2 Method A (Calibration with BBOT)—Repeatability and Reproducibility for Sulfur in 250 µm (No.60) Coal and Coke

	Range, %	Repeatability Limit, (r)	Reproducibility Limit (R)
Coal	0.37–5.48	$0.053 + 0.019 x^A$	$0.125 + 0.053 x^A$
Coke	0.6–6.71	$0.061 + 0.01 x^A$	$0.089 + 0.017 x^A$

[A] Where x is the average of two single test results.

TABLE 3 Method B-Repeatability and Reproducibility for Sulfur in 250 µm (No.60) Coal and Coke

	Range, %	Repeatability Limit (r)	Reproducibility Limit (R)
Coal and Coke[A]	0.38–5.6	$0.04 + 0.05x^B$	$0.07 + 0.07x^B$

[A] This precision statement is applicable to coal, metalurgical coke and petroleum coke.
[B] Where x is the average of two single test results.

ANNEX

A1. PRECISION STATISTICS

A1.1 The precision of this test method, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the following materials as listed in Table A1.1, Table A1.2, Table A1.3 and Table A1.4.

TABLE A1.1 Method A (Calibration with Coal and Coke Reference Materials)—Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters used for Calculation of Precision Statement of 250 μm (No.60) Coal

Material	Rank	Average	S_r	S_R	r	R
91–2	lvb	0.38775	0.011373	0.0248	0.031822	0.06939
91–1	hvAb	1.24325	0.014004	0.030672	0.039183	0.08554
91–5	hvAb	3.083	0.028988	0.060704	0.080879	0.16985
89–4	hvCb	5.6125	0.080452	0.209299	0.225106	0.58562
91–4	hvCb	0.27725	0.018384	0.025722	0.051439	0.07197
90–5	ligA	1.442	0.015066	0.044989	0.042155	0.12588
89–7	subA	0.75475	0.019184	0.028152	0.053677	0.07877
91–6	subA	0.46825	0.023578	0.021137	0.06597	0.05914
89–6	subC	0.5235	0.016505	0.023399	0.046433	0.06547
Lignite	lignite	0.666	0.021000	0.0415	0.058822	0.11605

TABLE A1.2 Method A (Calibration with BBOT)—Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters used for Calculation of Precision Statement of 250 μm (No.60) Coal[A]

Material[a]	Rank	Average	S_r	S_R	r	R
89–2	subB	0.3728	0.0193	0.0534	0.0541	0.1496
89–4	hvCb	5.4838	0.0598	0.1600	0.1675	0.4483
90–5	ligA	0.4570	0.0242	0.0657	0.0677	0.1839
89–7	subA	0.7904	0.0297	0.0723	0.0831	0.2023
89–8	hvBb	1.3698	0.0266	0.0453	0.0600	0.1270
89–9	hvAb	4.1013	0.0402	0.1035	0.1125	0.2899
90–1	subB	1.0095	0.0296	0.1176	0.0827	0.3292
91–2	lvb	0.3800	0.0192	0.0331	0.0536	0.0926
91–5	hvAb	3.0251	0.0385	0.1035	0.1079	0.2899
NIST 2682c	lvb	1.0828	0.0226	0.0410	0.0631	0.1149

[A]Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1042. Contact ASTM Customer Service at service@astm.org.

TABLE A1.3 Method A-Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters used for Calculation of Precision Statement of 250 μm (No.60) Coke[A]

Material	Coke Type	Average	S_r	S_R	r	R
Foundry	Met Coke	0.6939	0.0194	0.0360	0.0544	0.1009
Furnace	Met Coke	0.7960	0.0245	0.0437	0.0687	0.1224
DOE	Met Coke	0.6920	0.0148	0.0280	0.0415	0.0785
2017	Calcined Pet Coke	3.5154	0.0310	0.0668	0.0867	0.1860
3050	Green Pet Coke	4.0786	0.0350	0.0738	0.0979	0.2070
4070	Calcined Pet Coke	6.7059	0.0437	0.0638	0.1223	0.1764
5C19	Green Pet Coke	6.0084	0.0355	0.0705	0.0998	0.1875
7009	Met Coke	0.7136	0.0142	0.0326	0.0396	0.0911
NIST 2713a	Green Pet Coke	4.7735	0.0409	0.0630	0.1145	0.1764
NIST 2776	Met Coke	0.8478	0.0306	0.0367	0.0857	0.1027

[A]Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1042. Contact ASTM Customer Service at service@astm.org.

TABLE A1.4 Method B-Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters used for Calculation of Precision Statement of 250 μm (No.60) Coal and Coke[B]

Material	Average	S	S_r	S_R	r	R
Coal Bank 91-2	0.378833	0.013331	0.009134	0.015078	0.022776	0.042218
NIST 2693 Coal	0.4472	0.010438	0.014004	0.016007	0.039211	0.044804
NIST 2682b Coal	0.482708	0.016365	0.01825	0.022744	0.051101	0.063683
Lignite	1.16273	0.017295	0.060945	0.060945	0.170647	0.170647
NIST 2776 Coke	0.792708	0.00889	0.041456	0.041456	0.116106	0.116106
Metallurgical Coke	0.67025	0.013831	0.018783	0.021355	0.052609	0.059796
NIST 2719 Calcined Petroleum Coke	0.841556	0.015884	0.018552	0.022593	0.051947	0.06326
Coal Bank 91-1	1.213667	0.048008	0.027923	0.053754	0.078184	0.150512
Coal Bank 91-5	3.026083	0.070034	0.059552	0.087222	0.166774	0.244221
Coal Bank 89-4	5.630917	0.092162	0.124971	0.141506	0.349919	0.396216
AR 1713 Lignite	1.272167	0.117201	0.066552	0.130596	0.186346	0.365696
AR 2720 Green Petroleum Coke[A]						
AR 744 Calcined Petroleum Coke	2.383208	0.060416	0.076055	0.111863	0.212955	0.313216
NIST 2719 Green Petroleum Coke	4.563708	0.107464	0.081895	0.128758	0.229306	0.360521

[A] One of the Green Petroleum Coke samples used in this study was excluded from the statistics due to inconsistent results across all participating laboratories.
[B] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1041. Contact ASTM Customer Service at service@astm.org.

SUMMARY OF CHANGES

Committee D05 has identified the location of selected changes to this standard since the last issue (D4239–13) that may impact the use of this standard. (Approved March 1, 2014.)

(1) Subsections 1.4 and 6.6 added.

(2) Note 1 of subsection 8.2.2 revised.

Committee D05 has identified the location of selected changes to this standard since the last issue (D4239–12) that may impact the use of this standard. (Approved Oct. 1, 2013.)

(1) Subsections 3.1.1 and 7.5 added.
(2) Section 8 updated and subsection 8.2.2 added.
(3) Table 2 added and Section 11 updated.

(4) Tables A1.1 and A1.2 revised, Table A1.3 added, and previous Table A1.3 renumbered to A1.4.

Designation: D5865 – 13

Standard Test Method for
Gross Calorific Value of Coal and Coke[1]

This standard is issued under the fixed designation D5865; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method pertains to the determination of the gross calorific value of coal and coke by either an isoperibol or adiabatic bomb calorimeter.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific hazard statements are given in Section 8.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D121 Terminology of Coal and Coke
D346 Practice for Collection and Preparation of Coke Samples for Laboratory Analysis
D388 Classification of Coals by Rank
D1193 Specification for Reagent Water
D2013 Practice for Preparing Coal Samples for Analysis
D3173 Test Method for Moisture in the Analysis Sample of Coal and Coke
D3177 Test Methods for Total Sulfur in the Analysis Sample of Coal and Coke (Withdrawn 2012)
D3180 Practice for Calculating Coal and Coke Analyses from As-Determined to Different Bases
D4239 Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion

D7582 Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis
E144 Practice for Safe Use of Oxygen Combustion Vessels
E178 Practice for Dealing With Outlying Observations
E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory
E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Terminology

3.1 For additional definitions of terms used in this test method, refer to Terminology D121.

3.2 *Definitions:*

3.2.1 *adiabatic calorimeter*—a calorimeter that operates in the adiabatic mode and may or may not use a microprocessor. The initial temperature before initiating the combustion and the final temperatures are recorded by the operator or the microprocessor.

3.2.2 *automated calorimeter*—a calorimeter which has a microprocessor that takes the thermometric readings and calculates the Calibration Value and the Heat of Combustion Values.

3.2.3 *British thermal unit [Btu]*—is the amount of heat required to raise the temperature of one pound - mass [lbm] of liquid water at one atmosphere pressure one degree Fahrenheit at a stated temperature. The results of combustion calorimetric tests of fuels for steam power plants may be expressed in terms of the 1956 International Steam Table calorie (I.T. cal) which is defined by the relation, 1 I.T. cal = 4.1868 J. The Btu used in modern steam tables is defined by the means of the relation, 1 I.T. cal / g = 1.8 I.T. Btu / lb. Thus, 1 I.T. Btu / lb = 2.326 J / g.

3.2.4 *calorific value*—the heat produced by combustion of a unit quantity of a substance under specified conditions.

3.2.5 *calorimeter*—a device for measuring calorific value consisting of a bomb, its contents, a vessel for holding the bomb, temperature measuring devices, ignition leads, water, stirrer, and a jacket maintained at specified temperature conditions.

3.2.6 *gross calorific value (gross heat of combustion at constant volume), Q_v (gross)*—the heat produced by complete combustion of a substance at constant volume with all water formed condensed to a liquid.

3.2.7 *heat of formation*—the change in heat content resulting from the formation of 1 mole of a substance from its elements at constant pressure.

3.2.8 *isoperibol calorimeter*—a calorimeter that operates in the isoperibol mode and uses a microprocessor to record the initial and final temperatures and make the appropiate heat leak corrections during the temperature rise. It determines when the calorimeter is in equilibrium and ignites the sample and determines when the calorimeter has reached equilibrium after ignition.

3.2.9 *net calorific value (net heat of combustion at constant pressure), Q_v (net)*—the heat produced by combustion of a substance at a constant pressure of 0.1 MPa (1 atm), with any water formed remaining as vapor.

3.3 *Definitions of Terms Specific to This Standard:*

3.3.1 *corrected temperature rise*—the calorimeter temperature change caused by the process that occurs inside the bomb corrected for various effects.

3.3.2 *heat capacity*—the energy required to raise the temperature of the calorimeter one arbitrary unit.

3.3.2.1 *Discussion*—The heat capacity can also be referred to as the energy equivalent or water equivalent of the calorimeter.

4. Summary of Test Method

4.1 The heat capacity of the calorimeter is determined by burning a specified mass of benzoic acid in oxygen. A comparable amount of the analysis sample is burned under the same conditions in the calorimeter. The calorific value of the analysis sample is computed by multiplying the corrected temperature rise, adjusted for extraneous heat effects, by the heat capacity and dividing by the mass of the sample.

4.2 Oxidation of coal after sampling can result in a reduction of calorific value. In particular, lignite and sub-bituminous rank coal samples may experience greater oxidation effects than samples of higher rank coals. Unnecessary exposure of the samples to the air for the time of sampling or delay in analysis shall be avoided. (See X2.1.)

5. Significance and Use

5.1 The gross calorific value can be used to compute the total calorific content of the quantity of coal or coke represented by the sample for payment purposes.

5.2 The gross calorific value can be used for computing the calorific value versus sulfur content to determine whether the coal meets regulatory requirements for industrial fuels.

5.3 The gross calorific value can be used to evaluate the effectiveness of beneficiation processes.

5.4 The gross calorific value can be required to classify coals according to Classification D388.

6. Apparatus and Facilities

6.1 *Test Area*—An area free from drafts, shielded from direct sunlight and other radiation sources. Thermostatic control of room temperature and controlled relative humidity are desirable.

6.2 *Combustion Bomb*—Constructed of materials that are not affected by the combustion process or the products formed to introduce measurable heat input or alteration of end products. The bomb shall be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There shall be no gas leakage. The bomb shall be capable of withstanding a hydrostatic pressure test to 20 MPa (3000 psig) at room temperature without stressing any part beyond its specified elastic limit.

6.3 *Balance*—A laboratory balance capable of weighing the analysis sample to the nearest 0.0001 g. The balance shall be checked weekly, at a minimum, for accuracy.

6.4 *Calorimeter Vessel*—Made of metal with a tarnish-resistant coating, with all outer surfaces highly polished. Its size shall be such that the bomb is completely immersed in water during a determination. A stirrer shall be provided for uniform mixing of the water. The immersed portion of the stirrer shall be accessible to the outside through a coupler of low thermal conductivity. The stirrer speed shall remain constant to minimize any temperature variations due to stirring. Continuous stirring for 10 min shall not raise the calorimeter temperature more than 0.01°C when starting with identical temperatures in the calorimeter, test area and jacket. For calorimeters having a bucket it can be a separate component or integral component of the bomb. The vessel shall be of such construction that the environment of the calorimeter's entire outer boundaries can be maintained at a uniform temperature.

6.5 *Jacket*—A container with the inner perimeter maintained at constant temperature ±0.1°C (isoperibol) or at the same temperature ±0.1°C as the calorimeter vessel (adiabatic) during the test. To minimize convection the sides, top and bottom of the calorimeter vessel shall not be more than 10 mm from the inner surface of the jacket. Mechanical supports for the calorimeter vessel shall be of low thermal conductivity.

6.6 *Thermometers:*

6.6.1 *Platinum resistance or thermistor thermometers*—shall be capable of measuring to the nearest 0.0001°C. These types of thermometers consist of two major subsystems. The first and most obvious is the temperature sensing probe itself. The second and equally important aspect is the measurement subsystem. For both subsystems, the relationship between the thermometer resistance and temperature shall be well characterized. The absolute temperature shall be known to ±0.1 C at the temperature of the calorimetric measurement.

6.6.2 Assessing the valid working range of the calorimeter, as outlined in 10.9, is sufficient to demonstrate that all important aspects of the calorimeter functionality, including the thermometry, are in good working order. The traceability of the heat of combustion measurement is governed by the traceability of the heat of combustion of the benzoic acid calibrant in addition to meeting the aforementioned criteria.

6.6.3 *Liquid-in-Glass Thermometers*—Conforming to the requirements for thermometers S56C, S116C or S117C as prescribed in Specification E2251.

2

6.6.3.1 *Thermometer Accessories*—A magnifier is required for reading liquid-in-glass thermometers to one tenth of the smallest scale division. The magnifier shall have a lens and holder designed so as to minimize errors as a result of parallax.

6.7 *Sample Holder*—An open crucible of platinum, quartz, or base metal alloy. Before use in the calorimeter, heat treat base metal crucibles for a minimum of 4 h at 500°C to ensure the crucible surface is completely oxidized. Base metal alloy crucibles are acceptable, if after three preliminary firings, the weight does not change by more than 0.0001 g.

6.8 *Ignition Fuse*—Ignition fuse of 100-mm length and 0.16-mm (No. 34 B&S gauge) diameter or smaller. Nickel-chromium alloy (Chromel C) alloy, cotton thread, or iron wire are acceptable. Platinum or palladium wire, 0.10-mm diameter (No. 38 B&S gauge), can be used provided constant ignition energy is supplied. Use the same type and length (or mass) of ignition fuse for calorific value determinations as used for standardization.

6.9 *Ignition Circuit*—A 6- to 30-V alternating or direct current is required for ignition purposes. A step-down transformer connected to an alternating current circuit, capacitors, or batteries can be used. For manually operated calorimeters, the ignition circuit switch shall be of the momentary double-contact type, normally open except when held closed by the operator. An ammeter or pilot light can be used in the circuit to indicate when current is flowing.

6.10 *Controller*—For automated calorimeters, capable of charging the bomb, filling the calorimeter vessel, firing the ignition circuit, recording calorimeter temperatures before, during, and after the test, recording the balance weights, and carrying out all necessary corrections and calculations.

6.11 *Crucible Liner*—Quartz fiber or alundum for lining the crucible to promote complete combustion of samples that do not burn completely during the determination of the calorific value.[4]

7. Reagents

7.1 *Reagent Water*—Conforming to conductivity requirements for Type II of Specification D1193 for preparation of reagents and washing of the bomb interior.

7.2 *Purity of Reagents*—Use reagent grade chemicals conforming to the specification of the Committee on Analytical Reagents of the American Chemical Society in all tests.[5]

7.3 *Benzoic Acid—Standard (C_6H_5COOH)* Pellets made from benzoic acid available from the National Institute of Standards and Technology (NIST) or benzoic acid calibrated against NIST standard material. The calorific value of benzoic acid, for use in the calibration calculations, shall be traceable to a recognized certificate value.

[4] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1025.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.4 *Oxygen*—Manufactured from liquid air, guaranteed to be greater than 99.5 % pure, and free of combustible matter. Oxygen made by the electrolytic process contains small amounts of hydrogen rendering it unfit unless purified by passage over copper oxide at 500°C.

7.5 *Titration Indicator*—Methyl orange, methyl red, or methyl purple for indicating the end point when titrating the acid formed during combustion. The same indicator shall be used for both calibration and calorific value determinations.

7.6 *Standard Solution*—Sodium carbonate (Na_2CO_3) or other suitable standard solution. A convenient standard solution may be prepared as follows. Dissolve 3.706 g of sodium carbonate, dried for 24 h at 105°C, in water and dilute to 1 L. The resulting concentration (0.0699 N) assumes the energy of formation of HNO_3 under bomb conditions is -59.7 kJ/mol (-14.3 kcal/mole) (see X1.1). One milliliter of this solution is equivalent to 4.2 J (1.0 cal) in the acid titration. Alternatively, 1.0 ml of a 0.1000 N base solution is equivalent to 6.0 J (1.4 cal) in the acid titration. In general, one milliliter of an arbitrary standard titrant solution is equivalent to its concentration (equivalents / liter or normality [N]) times 59.7 J (14.3 cal).

8. Hazards

8.1 The following precautions are recommended for safe calorimeter operation. Additional precautions are noted in Practice E144. Also consult the calorimeter equipment manufacturer's installation and operating instructions before using the calorimeter.

8.1.1 The mass of sample and any combustion aid as well as the pressure of the oxygen admitted to the bomb shall not exceed the bomb manufacturer's specifications.

8.1.2 Inspect the bomb parts carefully after each use. Replace cracked or significantly worn parts. Replace O-rings and valve seats in accordance with manufacturer's instruction. For more details, consult the manufacturer.

8.1.3 Equip the oxygen supply cylinder with an approved type of safety device, such as a relief valve, in addition to the needle valve and pressure gauge used in regulating the oxygen feed to the bomb. Valves, gauges, and gaskets shall meet industry safety codes. Suitable reducing valves and adapters for 3- to 4-MPa (300- to 500-psig) discharge pressure can be obtained from commercial sources of compressed gas equipment. Check the pressure gauge annually for accuracy or after any accidental over pressures that reach maximum gauge pressure.

8.1.4 During ignition of a sample, the operator shall not extend any portion of the body over the calorimeter.

8.1.5 Do not fire the bomb if the bomb has been dropped or turned over after loading.

8.1.6 Do not fire the bomb if there is evidence of gas leakage when the bomb is submerged in the calorimeter vessel.

8.1.7 For manually operated calorimeters, the ignition switch shall be depressed only long enough to fire the charge.

9. Sample

9.1 The analysis sample is the material pulverized to pass 250-μm (No. 60) sieve, prepared in accordance with either Practice D346 for coke or Method D2013 for coal.

10. Determination of the Heat Capacity of the Calorimeter

10.1 *Sample*—Weigh 0.8 to 1.2 g of benzoic acid into a sample holder. Record sample weight to the nearest 0.0001 g.

10.2 *Preparation of Bomb*

10.2.1 Rinse the bomb with water to wet internal seals and surface areas of the bomb or precondition the calorimeter according to the manufacturer's instructions. Add 1.0 mL of water to the bomb before assembly.

10.2.2 Connect a measured fuse in accordance with manufacturer's guidelines.

10.2.3 Assemble the bomb. Admit oxygen to the bomb to a consistent pressure of between 2 and 3 MPa (20 and 30 atm). The same pressure is used for each heat capacity run. Control oxygen flow to the bomb so as not to blow material from the sample holder. If the pressure exceeds the specified pressure, detach the filling connection and exhaust the bomb. Discard the sample.

10.3 *Preparation of Calorimeter*

10.3.1 Fill the calorimeter vessel with water at a temperature not more than 2°C below room temperature and place the assembled bomb in the calorimeter. Check that no oxygen bubbles are leaking from the bomb. If there is evidence of leakage, remove and exhaust the bomb. Discard the sample.

10.3.2 The mass of water used for each test run shall be $M \pm 0.5$ g where M is a fixed mass of water. Devices used to supply the required mass of water on a volumetric basis shall be adjusted when necessary to compensate for change in the density of water with temperature.

10.3.3 With the calorimeter vessel positioned in the jacket start the stirrers.

10.4 *Temperature Observations Automated Calorimeters*

10.4.1 *Stabilization*—The calorimeter vessel's temperature shall remain stable over a period of 30 s before firing. The stability shall be ±0.001°C for an adiabatic calorimeters and ±0.001°C/s or less for an isoperibol calorimeter.

10.4.2 *Extrapolation Method*—Fire the charge, record the temperature rise. The test can be terminated when the observed thermal curve matches a thermal curve which allows extrapolation to a final temperature with a maximum uncertainty of ±0.002°C.

10.4.3 *Full Development Method*—Fire the charge and record the temperature rise until the temperature has stabilized for a period of 30 s in accordance with the stability requirements specified in 10.4.1.

10.5 *Temperature Observations Manual Calorimeters*

10.5.1 When using ASTM Thermometers S56C, estimate all readings to the nearest 0.002°C. When using ASTM Thermometers S116C, or S117C, estimate readings to 0.001°C and 25-Ω resistance thermometer readings to the nearest 0.0001 Ω. Tap or vibrate liquid-in-glass thermometers just before reading to avoid errors caused by liquid sticking to the walls of the capillary.

10.5.2 Allow 5 min for the temperature of the calorimeter vessel to stabilize. Adjust the jacket temperature to match the calorimeter vessel temperature within 0.01°C and maintain for 3 min.

10.5.3 Fire the charge. Record the time as a and the temperature as t_a.

10.5.4 For adiabatic calorimeters adjust the jacket temperature to match that of the calorimeter vessel temperature during the period of the rise. Keep the two temperatures as equal as possible during the period of rapid rise. Adjust to within 0.01°C when approaching the final stabilization temperature. Record subsequent readings at intervals no greater than 1 min until three successive readings do not differ by more than ±0.001°C. Record the first reading after the rate of change has stabilized as the final temperature t_c and the time of this reading as c. For isoperibol calorimeters, when approaching the final stabilization temperature, record readings until three successive readings do not differ by more than 0.001°C per min. Record the first reading after the rate of change has stabilized as the final temperature as t_c and the time of this reading as c.

10.5.5 *Bomb depressurization*—When the thermochemical corrections will be made by titration, the depressurization and subsequent rinse collection shall be done in a manner ensuring at least 80% recovery of the acidic combustion products. An effective measure of the recovery is offered by the recovery factor described in the following section.[6] Depressurization at a rate such that the operation is not less than one minute provides effective recovery and does not require validation. There are no special requirements related to the bomb depressurization if the calculated nitric acid procedure is used.

10.5.5.1 Determine the recovery factor from the slope of the straight line resulting from the regression analysis of the titration results (mg of sulfur vs. meq) using three or more samples with at least two different sulfur concentrations that span the range of sulfur values typically encountered in the laboratory. One equivalent of sulfuric acid (1000 meq) is formed from 16.03 grams of sulfur. A plot of sulfur (mg) vs. milliequivalents (meq) of titrant used to neutralize the bomb rinsings has a theoretical slope of 16.03. The milliequivalents of titrant is the product of milliliters of base used and its concentration (normality). The mass of sulfur in the sample in milligrams is the product of the sulfur concentration in weight percent in the analysis sample, the sample mass and ten. A slope of 16 implies 100% recovery. Increasing values are indicative of diminished recovery. For example, a value of 20 indicates 80% recovery (16.03 × 100 / 20 = 80%).

10.5.6 After depressurization, open the bomb and examine the bomb interior carefully for signs of unburned material or sooty deposits. Discard the test result if either is observed.

10.6 *Thermochemical Corrections (see Appendix X1)*

10.6.1 *Acid Correction (see X1.1)*—One may use either the titration (10.6.1.1) or calculated titration (10.6.1.2) procedure for coal and coke samples.

10.6.1.1 *Titration Method*—Wash the interior of the bomb with distilled water containing the titration indicator (see 7.5) until the washings are free of acid and combine with the rinse of the capsule. Titrate the washings with the standard solutions (see 7.6) using a titration indicator, or a pH or millivolt meter

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The product of the volume of titrant used in milliliters, the concentration of the standard solution (normality) and 59.7 J (14.3 cal) shall be taken as e1.

10.6.1.2 *Calculated Nitric Acid Method*—For test samples that contain no nitrogen, the nitric acid formed in the bomb during the combustion process is derived from the nitrogen in the air that occupies the bomb prior to pressurizing it with oxygen. The quantity of nitric acid formed is a function of the volume of the bomb, the oxygen filling pressure and the quantity of energy released in the bomb during a test. For a given bomb and filling pressure, the relationship between the amount of nitric acid formed and the energy released can be determined using the following procedure. The calorimeter should be tested using one-gram pellets of benzoic acid when the bomb volume is nominally 340 milliliters. If the bomb volume differs significantly from 340 ml, the sample weight used should be adjusted as required in order to comply with the bomb manufacturer's safe operating guidelines. Tests should be run in duplicate. Determine the nitric acid correction by titrating the bomb washings as outlined in 10.6.1.1. Divide the correction obtained (e1) by the amount of energy released in the bomb for the corresponding test. The energy contribution from the ignition fuse (e2) is usually small (<1% of the total energy released) and may be ignored. The average value of this ratio is used to establish a factor (NAF) that allows the nitric acid correction to be determined for any arbitrary energy release.

$$e1 = (NAF) * energy\ released\ for\ the\ test \qquad (1)$$

Example:

The nitric acid correction for a 340-ml oxygen bomb is found to be 41.9 J (10.0 calories) when the released energy corresponds to that liberated by one gram of benzoic acid, 26.45 kJ (6318 calories). The ratio of these quantities is 1.58 J per 1000 joules of released energy or 1.58 calories per 1000 calories of released energy. The nitric acid correction for any calorimetric test can then be expressed as follows.

$$e1 = 1.58 * energy\ released\ for\ the\ test\ (kJ\ or\ kcal) \qquad (2)$$

For a given calorimeter, the energy released in the bomb during a test for the purposes of calculating the nitric acid correction (e1) may be expressed as the product of the temperature rise for the test and the effective calorimeter heat capacity or calorimeter calibration factor.[6]

$$e1 = (1.58/1000) * E * t \qquad (3)$$

The calculated nitric acid method can be applied to samples containing up to 2% nitrogen without introducing a significant error in the resulting heat of combustion value.

10.6.2 *Fuse Correction (see X1.3)*—Determine the fuse correction using one of the two alternatives:

10.6.2.1 Measure the combined pieces of unburned ignition fuse and subtract from the original length to determine the fuse consumed in firing according to Eq 4.

$$e2 = K_l \times l \qquad (4)$$

where:

$e2$ = the correction for the heat of combustion of the firing fuse.

l = the length of fuse consumed during combustion,

K_l = 0.96 J/mm (0.23 cal/mm) for No. 34 B&S gauge Chromel C,

K_l = 1.13 J/mm (0.27 cal/mm) for No. 34 B&S gauge iron wire, and

K_l = 0.00 J/mm for platinum or palladium wire provided the ignition energy is constant.

or:

10.6.2.2 Weigh the combined pieces of unburned fuse and subtract from the original weight to determine the weight in milligrams of the fuse consumed in firing (m). Remove any ball of oxidized metal from the ends before weighing.

$$e2 = K_m \times m \qquad (5)$$

where:

$e2$ = the correction for the heat of combustion of the firing fuse,

m = the weight in mg of fuse consumed during combustion,

K_m = 5.9 J/mg (1.4 cal/mg) for No. 34 B&S gauge Chromel C,

K_m = 7.5 J/mg (1.8 cal/mg) for No. 34 B&S gauge iron wire, and

K_m = 0.00 J/mg for platinum or palladium wire provided the ignition energy is constant.

When cotton thread is used, employ the correction in J recommended by the instrument manufacturer.

10.7 *Calculation of the Corrected Temperature Rise*—Compute the corrected temperature rise, t, as follows:

$$t = t_c - t_o + C_c + C_r \qquad (6)$$

where:

t = corrected temperature rise, °C;

t_o = initial temperature reading at time of firing;

t_c = final temperature reading;

C_e = thermometer, emergent stem correction (see Eq A1.6 or Eq A1.8);

C_r = radiation correction (see Eq A1.1 or Eq A1.2).

10.7.1 The temperature rise in isoperibol calorimeters require a radiation correction.

10.8 *Calculation of the Heat Capacity*—Calculate the heat capacity (E) of the calorimeter using the following equation:

$$E = [(H_c \times m) + e1 + e2]/t \qquad (7)$$

where:

E = the calorimeter heat capacity, J/°C;

H_c = heat of combustion of benzoic acid, as stated in the certificate, J/g;

m = mass of benzoic acid, g;

$e1$ = acid correction from 10.6.1 from either the titration method (10.6.1.1) or the calculated titration (10.6.1.2);

$e2$ = fuse correction from 10.6.2, J; and

t = corrected temperature rise from 10.7, °C.

10.8.1 Using the procedures described in 10.1 – 10.8 complete a total of ten acceptable test runs. An individual test shall be rejected only if there is evidence of incomplete combustion.

10.8.2 The precision of ten acceptable calibration test runs shall have a relative standard deviation (RSD) no greater than 0.17%. If after considering the possibility of outliers using criteria established in Practice E178, this limit is not met, one should review operation of the calorimeter for any assignable cause. Operating deficiencies should be corrected before performing additional calibration measurements as required. Table 1 provides an example summary of a series of calibration tests. The formulas used to arrive at the mean value and relative standard deviation are independent of the units used for the calibration measurements.

10.9 *Valid Working Range for the Calibration of the Calorimeter:*

10.9.1 It is a generally accepted principle of reliable analysis that instruments should be calibrated over the full range of measurement and that measurements be restricted to the range calibrated. It is not good practice to report extrapolated data obtained outside the range of calibration. The range of reliable calibration can be considered as the range of reliable measurement and vice versa.

10.9.2 It should be possible to vary the amount of benzoic acid calibrant by at least ±25 % without observing a significant trend in the values obtained for the effective heat capacity or instrument calibration factor. The working limits for the instrument calibration shall be determined and defined in terms of the corrected temperature rise. All subsequent measurements shall be kept within these limits.

10.9.3 A convenient way to check a previously calibrated instrument is to use benzoic acid as an unknown. The mean value for the heat of combustion from duplicate runs using 0.7 g and 1.3 g sample masses, respectively, should be within ± 56 J/g or ±24 Btu / lb of the accepted heat of combustion value.

10.9.4 For any given instrument, examination of the applicable range of the instrument calibration should be carried out when the instrument is new, moved to a different location or subject to major repair.

11. Calibration Verification

11.1 The calorimeter calibration shall be checked on a regular basis. Renewed determination of the heat capacity is required whenever significant alterations have been made to the instrument or to the test conditions. The optimum frequency for checking the heat capacity or instrument calibration will depend on the stability of the measurement system and the risk involved when the system departs from statistical control. Since all data obtained during the period last-known-in-control to first-known-out-of-control are suspect, such intervals may need to be minimized.

11.1.1 There are several empirical approaches to deciding on how frequently the instrument calibration should be checked. The experience of the laboratory may indicate the expected frequency of occurrence of trouble, in which case reference sample measurements, at least three in number, should be equally spaced within such an interval. Another approach is the "length of run" concept. In this, recognizable breaks in the production (of data) process are identified which could cause significant changes in precision or bias. Such breaks could include change of work shift; rest periods; change, modification, or adjustment of apparatus; use of new calibration standards; significantly long down-times; use of a new lot of reagents. At least three reference samples should be measured during any of these periods when the periods are considered to be potentially significant.

11.1.2 Periodic checks of the instrument calibration are a risk-reducing procedure. However, if it involves more than ten percent of a laboratory's measurement effort, either the quality control process may need improvement or too much effort is being exerted in this direction. If less than five percent of effort is devoted to such measurements, the laboratory may be taking too high a risk of producing unacceptable data, or may not even know the quality of the data it is producing. The above statements are made with a laboratory making a significant number of high-quality routine measurements in mind. If a laboratory's program involves occasional or one-of-a-kind

TABLE 1 Calibration Measurements

Run Number	Column A Heat Capacity J/°C	Column B Difference from Average Squared
1	10258	20
2	10249	20
3	10270	272
4	10254	0
5	10246	72
6	10249	20
7	10241	156
8	10266	156
9	10258	20
10	10245	72
Sum	102536	811
Average	10254	Sum Column A /10
Variance (s²)	90	Sum Column B /9
Standard Deviation (s)	9	Sqrt (Variance)
Relative Standard Deviation (rsd (%))	0.09%	s/Average * 100

measurements, the amount of quality assurance effort required, including the number of measurements of reference materials to be made may be significantly more than that indicated above.

11.1.3 Two complementary procedures are offered for calorimeter calibration verification: Control Chart Method and Rolling Average Method.

11.2 *Control Chart Method for Calibration Verification:*

11.2.1 A control chart is a graphical way to interpret test data. In its simplest form, a selected reference sample is measured periodically and the results are plotted sequentially (or time-ordered) on a graph. Limits for acceptable values are defined and the measurement system is assumed to be in control (variability is stable and due to chance alone) as long as the results stay within these limits. The residence of the values within expected limits is accepted as evidence that the precision of measurement remains in control. The monitored precision of measurement and the accuracy of measurement of the reference sample may be transferred, by inference, to all other appropriate measurements made by the system while it is in a state of control.

11.2.2 A control chart can be realized by sequentially plotting individual measurement values. The central line is the most probable value for (that is, the grand average) of all the measurements or the accepted measurement value. The limits LWL to UWL (lower and upper warning limits) define the area in which 95 percent of the plotted points are expected to lie. The limits LCL to UCL (lower and upper control limits) define the area in which almost all (99.7%) of the plotted points are expected to lie when the system is in a state of statistical control. It should be clear that when more than 5 percent of the points (one in twenty) lie outside of the warning limits or when values fall outside of the control limits the system is behaving unexpectedly and corrective actions, and even rejection of data, may be required.

11.2.3 Results are expected to scatter with a normal distribution within the limits. Systematic trends or patterns in the data plots may be early warning of incipient problems and are cause for concern; hence techniques to identify such should be practiced.

11.2.4 Control charts, including the factors for calculating control limits are discussed more thoroughly elsewhere (see Guide E882 and (1)[5]. The central line is either the known value for the test sample (for example, certified value), or the mean of 10 sets of independent measurements. Control limits are then calculated according to the following relationships:

UCL	Mean or accepted value + 3 * sigma / sqrt(N)
UWL	Mean or accepted value + 2 * sigma / sqrt(N)
Central Line:	Mean of the 10 most recent measurements or the
X(bar)	accepted value.
LWL	Mean or accepted value - 2 * sigma / sqrt(N);
LCL	Mean or accepted value - 3 * sigma / sqrt(N)

For the above limits, N represents the number of repetitive measurements of the reference sample, the mean of which is plotted on an Xbar chart. For an X chart (single measurement of the reference sample) N = 1. The standard deviation of the measurement process is sigma. Sigma is taken as 0.10% of the mean of the ten most recent measurements or 0.10% the accepted value if tests are being performed using a reference material, for example benzoic acid, run as an unknown. Table 2 illustrates important selected control limits for calorimeter operation when benzoic acid is used as a test sample.

11.3 *Rolling Average Method:*

11.3.1 The mean value of the ten most recent calorimeter calibration or heat capacity measurements is used to support the calibration of the calorimeter system. A single new value, incorporated into the average of the ten most recent measurements, discarding the oldest measurement in the existing series of ten measurements first, must meet the precision requirements outlined in 10.8.2.

11.4 *Recommended Test Substances.*

[5] The boldface numbers in parentheses refer to a list of references at the end of this standard.

TABLE 2 Calorimeter Control Limits When Benzoic Acid is Used as a Test Sample

Note 1—Accepted heat of combustion taken as 26454 J/g
Process sigma is 0.10% RSD
Control limits based on 99% confidence (3 sigma) values
Values are in J/g except as noted.

Number of Observations in a Group	UCL for the range (High-Low) within the Group	UCL for the RSD within the Group	Maximum Permissible Deviation of the Group Mean from the Accepted Value or Grand Mean
1	...		79.4
2	97.5	0.2606	56.1
3	115.3	0.2275	45.8
4	124.3	0.2088	39.7
5	130.1	0.1954	35.5
6	134.3	0.1874	32.4
7	137.6	0.1806	30.0
8	140.4	0.1751	28.1
9	142.7	0.1707	26.5
10	144.7	0.1669	25.1
15	151.9	0.1554	20.5
20	156.7	0.1470	17.7
25	160.2	0.1470	15.9

Calorimeter Heat Capacity X Chart
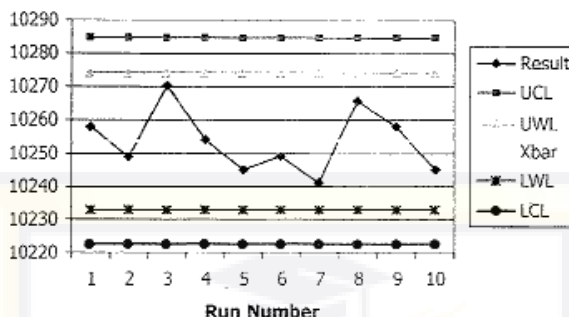
FIG. 1 Example of X Chart for Calibration Runs

Benzoic Acid Check Sample X Chart
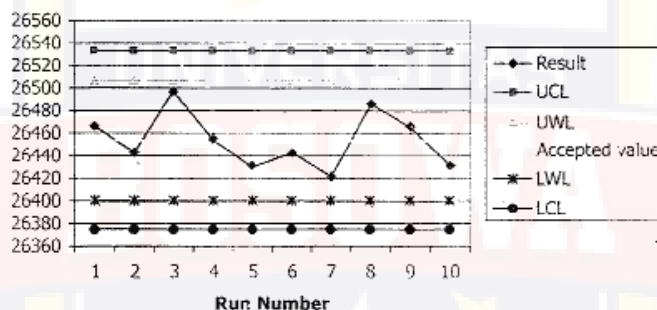
FIG. 2 Example of X Chart for Check Sample Runs

11.4.1 By international agreement in the 1920's, benzoic acid was selected as the chemical standard to be used in calibrating bomb calorimeters. Today, it remains as the sole chemical primary standard for this type of calibration.

11.4.1.1 In general, the calibration of an analysis instrument consists of the evaluation of its response function in terms of the composition of the analyte. The instrument responds to some property of the analyte, the value of which needs to be quantified by use of known substances. It is tacitly assumed that the instrument will respond analogously to the standard and test samples. With this in mind, the confidence in the measurement process is influenced by the uncertainty in composition of known samples and the soundness of the analogy.

11.4.1.2 It is desirable to perform check experiments in order to demonstrate confidence in the ability to make measurements on unknowns that are not only acceptably precise but also acceptably accurate. This is especially true in instances where conditions in the calibration and combustion tests differ appreciably. For example, large differences in the rates of combustion between the calibrant and the unknown or significant differences in combustion chemistries may give rise to systematic errors that are difficult to detect. The use of test substances in such check experiments can help demonstrate that the techniques and procedures used in determining heats of combustion of unknown materials are reasonably free of persistent errors.

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11.4.2 For work with solid samples, the following test substances are recommended.

Substance	$-\Delta H_g$ / Jg[-1][A]
Acetanilide	31279 (2)
CAS 103-84-4	
CH3CONHC6H5	
MWT 135.17	
Density 1.22 g/cc	
Tris (hydroxymethyl) aminomethane	20058 (3)
CAS 77-86-1	
(HOCH2)3-CNH2	
MWT 121.14	
Density = 1.35 g/cc	

[A] Mass against stainless steel weights (8 g/cc) in air (0.0012 g/cc).

11.4.2.1 Both acetanilide and tris (hydroxymethyl) aminomethane are available in high purity, are non-hygroscopic and have very low vapor pressure. Acetanilide is available from NIST as a micro-analytical standard for CHON compounds (SRM 141). Tris (hydroxymethyl) aminomethane is also available from NIST as an acidimetric standard (SRM 723).

11.4.2.2 Both materials are commercially available. When the aforementioned materials are used as secondary thermo-chemical standards or test substances for combustion calorimetry, ensure that the minimum stated purity is at least 99.9%.

11.4.2.3 Crystalline tris (hydroxymethyl) aminomethane is difficult to form into a durable pellet. Its relatively low heat of combustion value allows this material to be weighed directly into the combustion crucible and combusted in this form. When a consumable metal fuse wire is used, the wire loop shall be in direct contact with the granular sample.

11.4.2.4 Acetanilide is easily formed into a 12.5 mm diameter pellet and shall be burned in this form due to its higher heat of combustion value.

11.4.2.5 The nitric acid correction for tests involving the recommended test substances is approximately 50% higher than what's typically obtained for benzoic acid calibrant samples. This is a consequence of the single nitrogen atom associated with each of the test materials. About 10% of this nitrogen gives rise to aqueous nitric acid during the high pressure combustion process. The remainder appears as elemental nitrogen in the combustion products. The additional correction is accounted for transparently when the titration method is used to arrive at the acid correction. When the calculated nitric acid method is used, the nitric acid factor (NAF) shall be increased by 50% when applied to tests involving these test substances.

11.5 Use of Laboratory Control Materials:

11.5.1 A laboratory control material (LCM) is similar to a certified reference material in that it is a homogeneous matrix that offers key characteristics similar to the samples being analyzed. A true LCM is one that is prepared and stored in a stable condition strictly for use in-house by a single laboratory. Alternately, the material may be prepared by a central laboratory and distributed to others (so-called regional or program control materials). Unlike CRMs, the heat of combustion of LCMs are not certified but are based upon a statistically valid number of replicate analyses by one or several laboratories. In practice, this material can be used to assess the performance of

a single laboratory, as well as to determine the degree of comparability among different laboratories.

11.5.2 A significant amount of work is involved with the production and maintenance of any LCM. For example, materials related issues such as stability, homogenization techniques and proper conditions for storage must be addressed. For smaller laboratories, the time and effort involved supporting the use of true LCMs may not be justified. In such cases, the use of CRMs is preferred.

11.5.3 Routine analysis of CRMs or LCMs represent an important aspect of a performance-based QA philosophy. At least one CRM or LCM must be analyzed along with each batch of 20 or fewer samples (that is, QA samples should comprise a minimum of 5% of each set of samples). For CRMs, the certified heat of combustion value will be known to the analyst(s) and will be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy must be established for the analysis of CRMs or LCMs using a given instrumental technique. If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM or LCM, the data for the entire batch of samples is suspect. Potential operating deficiencies must be checked / corrected and the CRM or LCM may have to be reanalyzed to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to find and eliminate the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before final data are reported.

12. Procedure for Coal and Coke Samples

12.1 Weigh 0.8 to 1.2 g of sample into a sample holder. Record the weight to the nearest 0.0001 g (see 12.6.1).

12.2 Follow the procedures as described in 10.2 – 10.5 for determination of heat capacity. For the calorific value of coke, it is necessary to use 3-MPa (30-atm) pressure for both standardization and analysis. The starting temperature for determinations shall be within ±0.5°C of that used in the determination of the heat capacity.

12.2.1 For coke, place a clean combustion capsule in the center of a quartz disk and press the capsule to make an impression in the disk. Cut slits from the outside edge of the disk to the impression. Insert the quartz disk in the combustion capsule so that the slit portion will cover the sides of the capsules.

12.3 Carry out a moisture determination in accordance with Test Method D3173 or Test Methods D7582 on a separate portion of the analysis sample preferably on the same day but not more than 24 h apart from the calorific value determination so that reliable corrections to other bases can be made.

12.4 Conduct the sulfur analysis in accordance with Test Methods D3177 or D4239. From the weight % sulfur, calculate the sulfur corrections (see X1.2).

12.4.1 When titration method is used (see 10.6.1.1), the sulfur correction is

$$e_3 = 55.5 \text{ J/g} \times S \times m \text{ or } (13.3 \text{ cal/g} \times S \times m) \qquad (8)$$

where:

$e3$ = a correction for the difference between the heat of formation of H_2SO_4 from SO_2 with respect to the formation of HNO_3, J;

S = wt % sulfur in the sample; and

m = mass of sample from 12.1, g.

12.4.2 When the calculated nitric acid method is used (see 10.6.1.2), the sulfur correction is

$$e3 = 92.7\ J/g \times S \times m\ \text{or}\ (22.1\ cal/g \times S \times m) \qquad (9)$$

where:

$e3$ = a correction associated with the heat of formation of H_2SO_4 from SO_2, J;

S = wt % sulfur in the sample, and

m = mass of sample from 12.1, g.

12.5 For eight mesh samples, analyze coals susceptible to oxidation within 24 h of preparation.

12.6 Coal or coke that do not burn completely can be treated as follows:

12.6.1 For coke, use a crucible liner of the type recommended in 6.11.

12.6.2 Use a combustion aid such as benzoic acid, ethylene glycol, mineral oil or a gelatin capsule. A minimum of 0.4 g of combustion aid shall be used. Record the weight to the nearest 0.0001 g. Calculate the correction for use of a combustion aid using the following:

$$e4 = Ha \times ma \qquad (10)$$

where:

$e4$ = correction for use of a combustion aid,

Ha = heat of combustion of the combustion aid J/g (cal/g), and

ma = mass of combustion aid, g.

12.6.3 Vary the mass of the sample to obtain complete combustion while confining the temperature rise within the valid working range of calibration.

13. Calculation

13.1 *Gross Calorific Value*—Calculate the gross calorific value $Q_{vad}(gross)$ using the following equation:

$$Q_{vad}(gross) = [(tE_c) - e1 - e2 - e3 - e4]/m \qquad (11)$$

where:

$Q_{vad}(gross)$ = gross calorific value at constant volume as determined, J/g (cal/g);

E_c = the heat capacity of the calorimeter, J/°C (cal/°C),

t = corrected temperature rise according to 10.7, °C;

$e1$ = acid correction according to 10.6.1, J;

$e2$ = fuse correction according to 10.6.2, J;

$e3$ = sulfur correction determined according to 13.4, J;

$e4$ = combustion aid correction determined according to 12.6.2, J; and

m = mass of the sample, g.

13.1.1 See X1.5 for an example calculation.

13.2 *Net Calorific Value at Constant Pressure:*

13.2.1 Several steps are required in order to report an as-received net heat of combustion, at constant pressure, derived from an as determined gross heat of combustion at constant volume. The first step involves calculating a gross heat of combustion at constant pressure from the calorimetrically determined gross heat of combustion in the bomb at constant volume.

13.2.2 No work is performed in constant-volume bomb calorimetry, so the heat measured equals the change in internal energy of the system. When fuel is burned at constant pressure, there is a change in the volume of the system. A small change in energy accompanies this change in volume. When fuel is burned at constant pressure and the water formed condensed to the liquid state, there is a contraction in the volume of the system. This contraction is equal to the volume of oxygen required to burn the hydrogen. Work is done on the system by the atmosphere in filling this void in order to maintain a constant pressure. When carbon in the fuel reacts with oxygen, an equal volume of carbon dioxide results and no change in volume occurs. The oxygen and nitrogen in the fuel both give rise to an increase in volume. The energy associated with this change in the volume of the gaseous phase for the combustion reaction may be expressed as follows.

$$Qr = p = 0.01 * RT * (H_{ad}/(2 * 2.016) - O_{ad}/31.9988 - N_{ad}/28.0134) \qquad (12)$$

R is the universal gas constant (8.3143 J/(mol *K)) and T is the standard thermochemical reference temperature (298.15 K).

13.2.2.1 H_{ad}, O_{ad}, and N_{ad} are as-determined hydrogen, oxygen and nitrogen, respectively, %, in the analysis sample. The hydrogen and oxygen contributed by the sample moisture are not included in H_{ad} and O_{ad}, respectively.

13.2.2.2 The following formulas may be used to convert hydrogen and oxygen values that include the hydrogen and oxygen in the moisture associated with the analysis sample to values excluding the moisture.

$$H_{ad} = H_{adm} - 0.1119 * M_{ad} \qquad (13)$$

$$O_{ad} = O_{adm} - 0.8881 * M_{ad} \qquad (14)$$

13.2.3 The next steps involve calculating the energy associated with the heat of vaporization of water that originates from the hydrogen content of the sample [Qh], the moisture content of the analysis sample [Qmad] as well as the as-received moisture value [Qmar].

$$Qh = 0.01 * Hvap * (Had/2.016) \qquad (15)$$

Hvap is the constant pressure heat of vaporization of water at 25 C (43985 J/mol)

$$Qmad = 0.01 * Hvap * (Mad/18.0154) \qquad (16)$$

$$Qmar = 0.01 * Hvap * (Mar/18.0154) \qquad (17)$$

Mad and Mar are values for moisture as-determined and as-received, respectively in weight %.

13.2.4 The as-determined net heat of combustion at constant pressure is equal to calorimetrically determined heat of combustion with the addition of the small constant volume to constant pressure correction, less the energy associated with

the latent heat of vaporization of water originating from the fuel hydrogen and the sample moisture.

$$Qpad(net) = Qvad(gross) + Qv - p - Qh - Qmad \qquad (18)$$

13.2.5 The dry net heat of combustion at constant pressure is given by the following relationship.

$$Qpd(net) = \{Qvad(gross) + Qv - p - Qh\}*\{100/(100 - Mad)\} \qquad (19)$$

13.2.6 The as-received net heat of combustion at constant pressure is given by the following relationship.

$$Qpar(net) = \{Qvad(gross) + Qv - p - Qh\}*\{100 - Mar)/(100 - Mad)\} - Qmar \qquad (20)$$

13.2.7 For reference purposes, the dry and as-received gross heat of combustion values at constant volume are provided by the following well-known relationships described in Practice D3180.

$$Qvd(gross) = Qvad(gross)*\{100/(100 - Mad)\} \qquad (21)$$

$$Qvar(gross) = Qvad(gross)*\{100 - Mar)/(100 - Mad)\} \qquad (22)$$

Example:

Qvad(gross)	30000 J/g
Mad	2.00%
Mar	8.00%
H(ad)	4.00%
O(ad)	13.00%
N(ad)	1.00%
Qv-p	13.6 J/g
Qh	872.7 J/g
Qmad	48.8 J/g
Qmar	195.3 J/g
Qvar	28163.3 J/g
Qpad(net)	29092.1 J/g
Qpd(net)	29735.6 J/g
Qpar(net)	27161.6 J/g

13.3 *Net Calorific Value at Constant Pressure using Dry Basis Values:*

13.3.1 The energy associated with the change in volume of the gaseous phase for the combustion reaction is expressed as follows:

$$Q_{v-p} = 0.01*RT*\{H_d/(2*2.016) - O_d/31.9988 - N_d/28.0134\} \qquad (23)$$

where:

R = the universal gas constant [8.3143 J/(mol *K)] and T is the standard thermochemical reference temperature (298.15 K).

$H_d, O_d, \text{ and } N_d$ = dry basis hydrogen, oxygen and nitrogen respectively, %, in the analysis sample.

13.3.2 The next steps involve calculating the energy associated with the heat of vaporization of water that originates from the hydrogen content of the sample $\{Q_h\}$, and the as-received moisture value $\{Q_{mar}\}$.

$$Q_h = 0.01*H_{ad}*(H_g/2.016) \qquad (24)$$

H_{vap} = the constant pressure heat of vaporization of water at 25°C (43985 J/mol)

$$Q_{mar} = 0.01*H_{vap}*(M)_{ar}/18.0154) \qquad (25)$$

M_{ar} = moisture as-received in weight %

13.3.3 The dry net heat of combustion at constant pressure is equal to the dry basis calorimetrically determined heat of combustion with the addition of the small constant volume to constant pressure correction, less the energy associated with the latent heat of vaporization of water originating from the fuel hydrogen.

$$Q_{pd}(net) = Q_{vd}(gross) + Q_{v-p} - Q_h \qquad (26)$$

13.3.4 The as-received net heat of combustion at constant pressure is given by the following relationship.

$$Q_{par}(net) = \{Q_{vd}(gross) + Q_{v-p} - Q_h\}*\{(100 - M_{ar})/100\} - Q_{mar} \qquad (27)$$

13.3.5 For reference purposes, the dry and as-received gross heat of combustion values at constant volume are provided by the following well-known relationships described in Practice D3180.

$$Q_{vd}(gross) = Q_{vad}(gross)*\{100/(100 - M_{ad})\} \qquad (28)$$

$$Q_{var}(gross) = Q_{vd}(gross)*(100 - M_{ar})/100 \qquad (29)$$

M_{ad} = moisture as-determined in weight %

Example:

Qvd(gross)	30612 J/g
M_ar	8.00%
H_d	4.08%
O_d	13.27%
N_d	1.02%
Qv-p	13.9 J/g
Q_h	890.2 J/g
Q_mar	195.3 J/g
Q_var (gross)	28163.0 J/g
Q_pd(net)	29735.7 J/g
Q_par(net)	27161.6 J/g

14. Report

14.1 Report the calorific value as Q_{vad} (gross) along with the moisture of the sample as determined M_{ad} from 12.3.

14.2 The results of the calorific value can be reported in any of a number of bases differing in the manner the moisture is treated. Procedures for converting the value obtained on an analysis sample to other bases are described in Practice D3180.

15. Precision and Bias[6]

15.1 *Precision*

15.1.1 *Manual Adiabatic Calorimeters*

15.1.1.1 *250 μm (No. 60) Samples:*

(1) Repeatability Limit (r)—The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) of separate and consecutive test determinations, carried out on the same sample of 250 μm (No. 60) coal and coke in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %. The repeatability limit for this test method is 115 J/g (50 Btu/lb).

(2) Reproducibility Limit (R)—The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) carried out in different laboratories

using samples of 250 µm (No. 60) coal and coke taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %. The reproducibility limit for this test is 250 J/g (100 Btu/lb).

15.1.1.2 *2.36 mm (No. 8) Samples:*[9]

(1) Repeatability Limit (r)— The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) of separate and consecutive test determinations, carried out on the same sample, using the same riffle, determined on a single test specimen of two separate 2.36 mm (No. 8) test units of coal reduced entirely to 250 µm (No. 60) and prepared from the same bulk sample coal in the same laboratory by the same operator using the same apparatus on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

TABLE 3 Ranges and Limits of Repeatability for the Gross Calorific Value of 2.36 mm (No.8) Coal with Manual Adiabatic Calorimeters

Coal	Range	Repeatability Limit (r)
Bituminous	29 535 to 33 720 J/g (12 700 to 14 600 Btu/lb)	160 J/g (69 Btu/lb)
Subbituminous-Lignite	20 442 to 29 651 J/g (8790 to 12 750 Btu/lb)	140 J/g (59 Btu/lb)

(2) Reproducibility Limit (R) –The value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) carried out in different laboratories using samples of 2.36 mm (No. 8) coal reduced entirely to 250 µm (No. 60) taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

TABLE 4 Ranges and Limits of Reproducibility for the Gross Calorific Value of 2.36 mm (No.8) Coal with Manual Adiabatic Calorimeters

Coal	Range	Reproducibility Limit (R)
Bituminous	29 535 to 33 720 J/g (12 700 to 14 600 Btu/lb)	249 J/g (107 Btu/lb)
Subbituminous-Lignite	20 442 to 29 651 J/g (8790 to 12 750 Btu/lb)	326 J/g (140 Btu/lb)

15.1.1.3 Precision statements for 2.36 mm (No. 8 sieve) samples have not been developed for anthracite coals.

15.1.2 *Automated Calorimeters:*

15.1.2.1 *Microprocessor Controlled Calorimeters:*[9],[10]

[9] An interlaboratory study, designed consistent with Practice E691, was conducted in 1989. Eight laboratories participated in this study. Supporting data are available from ASTM Headquarters. Request Report RR:D05-1015.
[10] An interlaboratory study, designed consistent with Practice E691, was conducted in 1995. Twelve labs participated in this study. Supporting data are available from ASTM Headquarters. Request Report RR:D05-1020.

TABLE 5 Ranges and Limits of Repeatability for the Gross Calorific Value of 250 µm (No. 60) Coal and Coke with Microprocessor Controlled Calorimeters

Material	Range	Repeatability Limit (r)
Coke	Nominally 30340 J/g (13 040 Btu/lb)	125 J/g (54 Btu/lb)
Bituminous	26 280 to 34 190 J/g (11 300 to 14 700 Btu/lb)	149 J/g (64 Btu/lb)
Subbituminous-Lignite	21 860 to 27 680 J/g (9400 to 11 900 Btu/lb)	193 J/g (83 Btu/lb)

TABLE 6 Ranges and Limits of Reproducibility for the Gross Calorific Value of 250 µm (No. 60) Coal and Coke with Microprocessor Controlled Calorimeters

Material	Range	Reproducibility Limit (R)
Coke	Nominally 30340 J/g (13 040 Btu/lb)	258 J/g (111 Btu/lb)
Bituminous	26 280 to 34 190 J/g (11 300 to 14 700 Btu/lb)	256 J/g (110 Btu/lb)
Subbituminous-Lignite	21 860 to 27 680 J/g (9400 to 11 900 Btu/lb)	381 J/g (164 Btu/lb)

(1) Precision—The precision of this method for the determination of calorific value in the analysis sample of coal and coke is shown in Tables 5 and 6. The precision characterized by repeatability (S_r, r) and reproducibility (S_R, R) is described in Table A2.1 and Table A2.2 in Annex A2.

(2) Repeatability Limit (r)— 250 µm (No. 60) Samples-the value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) of separate and consecutive test determinations, carried out on the same sample of 250 µm (No. 60) coal and coke in the same laboratory by the same operator using the same apparatus with the same heat capacity on samples taken at random from a single quantity of homogeneous material, may be expected to occur with a probability of approximately 95 %.

(3) Reproducibility Limit (R)—250 µm (No. 60) Samples-the value below which the absolute difference between two test results calculated to a dry basis (Practice D3180) carried out in different laboratories using samples of 250 µm (No. 60) coal and coke taken at random from a single quantity of material that is as homogeneous as possible, may be expected to occur with a probability of approximately 95 %.

15.1.2.2 Precision statements have not been developed for 2.36 mm (No. 8 sieve) size coal samples or for 250 µm (No. 60) anthracite coals.

15.2 *Bias*—Bias in the determination of the gross calorific value is eliminated provided samples are treated identically to the benzoic acid used in the determination of the calorimeter heat capacity.

15.3 The net heat of combustion result and values calculated to other reporting bases are affected by errors incurred in all the determinations used to evaluate the value. As a result, the repeatability and reproducibility limits set forth in this section are only applicable to dry gross heat of combustion values.

16. Keywords

16.1 adiabatic calorimeter; bomb calorimeters; calorific value; calorimeter; coal; coke; isoperibol bomb calorimeter

ANNEXES

(Mandatory Information)

A1. THERMOMETRIC CORRECTIONS

A1.1 *Thermometer Corrections*—The following corrections shall be made:

A1.1.1 *Calibration Correction*, shall be made in accordance with the calibration certificate furnished by the calibration authority.

A1.1.2 *Radiation Corrections*—Radiation corrections are required to calculate heat loss or gain to the isoperibol water jacket. They are based on the Dickinson formula,(4) the Regnault-Pfaundler formula,(5) or the U.S. Bureau of Mines method.(6) The same method of determining the radiation correction shall be used consistently in the determination of heat capacity and sample measurements.

A1.1.2.1 *Dickinson Formula:*

$$C_r = -r_1(b - a) - r_2(c - b) \qquad (A1.1)$$

where:

C_r = radiation correction;
r_1 = rate of rise in temperature per minute in the preliminary period. If temperature is falling, r_1 is negative;
r_2 = rate of rise of temperature per minute in the final period. If temperature is falling, r_2 is negative;
t_o = firing temperature;
t_f = final temperature, being the first temperature after which the rate of change is constant;
a = time at temperature t_o;
b = time at temperature $t_o + 0.60 \, (t_f - t_o)$; and
c = time at temperature t_f.

A1.1.2.2 Regnault-Pfaundler Formula:

$$C_r = (a - a)[k(t_a - t) - r_1 \qquad (A1.2)$$

where:

C_r = radiation correction,

$$k = (r_1 - r_2)/(t'' - t') \qquad (A1.3)$$

$$t_a = 1/n \left(\sum_{i=1}^{i} t_i + (t_o + t_a)/2 \right) \qquad (A1.4)$$

n = the number of temperature readings in the combustion period after firing;
t' = average temperature during the preliminary period;
t'' = average temperature during the final period;
$t_0, t_2 \cdots t_n$ = successive temperature recorded during the combustion period after firing at equal time intervals (for example, 1 min.) and

$\sum_{i=1}^{i} t_i =$ the sum of $t_1, t_2, t_3 \cdots t_{n-1}$

The remaining symbols are the same as defined in the Dickinson formula.

A1.1.2.3 *Bureau of Mines Method*—A table of radiation corrections can be established so that only the initial and final readings are required to determine the calorific value of a fuel. This can be done by carrying out a series of tests using the procedure described in Section 10, using the following conditions. Regulate the amount of sample burned so that a series of determinations is made in which different temperature rises are obtained. For all determinations, keep the water jacket temperature constant, fire the bomb at the same initial temperature, and have the same time, $c - a$, elapse (± 2 s) between the initial and final readings. Determine the radiation corrections for each of the series of temperature rises using the Dickinson method (see A1.1.2.1), or the Regnault-Pfaundler method (see A1.1.2.2). These corrections are constant for a given temperature rise. From the series of readings, a table or graph is plotted to show radiation correction versus temperature rise. Once the table or graph is established, the radiation corrections can be obtained from it until there is a major change in the equipment.

A1.1.2.4 *Example*—See Table A1.1.

A1.1.3 *Emergent Stem Correction*—
The calculation of emergent stem correction depends upon the way the thermometer was calibrated and how it is used. Two conditions are possible.

TABLE A1.1 Observed Calorimeter Temperature Readings

Time	Temperature	
0 (start)	22.4132	t_{start}
1	22.4207	
2	22.4284	
3	22.4361	
4	22.4436	
5 (a)	22.4512	t_a
6 (b)	23.9350	
7	24.4234	
8	24.5763	
9	24.6300	
10	24.6515	
11	24.5612	
12	24.6664	
13	24.6688	
14	24.6725	
15 (c)	24.6750	t_c
16	24.6773	
17	24.6795	
18	24.6816	
19	24.6838	
20 (end)	24.6860	t_{end}

Calculation of the Radiation Correction and the Corrected Temperature Rise (t)

$r_i = (t_a - t_{start}) / (a - \text{start})$ = (22.4512 − 22.4132) / 5 = 0.0076
$r_2 = (t_{end} - t_c) / (\text{end} - c)$ = (24.6860 − 24.6750) / 5 = 0.0022
$t'' = (t_a + t_{start}) / 2$ = (22.4512 + 22.4132) / 2 = 22.4322
$t = (t_{end} + t_c) / 2$ = (24.6860 + 24.6750) / 2 = 24.6805
$k = (r_1 + r_2) / (t'' − t')$ = 0.0024
$t_m = 1/n (\sum_{i=1}^{n} t_i + (t_a + t_c) / 2)$ = 1/10 (220.7861 + 23.5631) = 24.4349
C_r (Dickinson) = $r_1 (b − a) − r_2 (c − b)$ = − 0.0076 (1) − 0.0022 (9) = − 0.0274
C_r (Regnault-Pfaundler) = $(c − a) (k (t_m − t_1 − t_2))$ = (15 − 5) (0.0024) (24.4349 − 22.4822) − 0.0076) = − 0.0279
$t_{Dickinson} = t_c − t_a + C_r$ = (24.6750 − 22.4512) − 0.0274 = 2.1964
$t_{Regnault-Pfaundler} = t_c − t_a + C_r$ = (24.6750 − 22.4512) − 0.0279 = 2.1959

A1.1.3.1 *Thermometers Calibrated in Total Immersion and Used in Partial Immersion*—This emergent stem correction is made as follows:

$$C_e = K(t_f − t_i)(t_f + t_i − L − T) \quad (A1.5)$$

where:

C_e = emergent stem correction,
K = differential coefficient of thermal expansion for the thermometric liquid, (See Note A1.1.)
L = scale reading to which the thermometer was immersed,
T = mean temperature of emergent stem,
t_i = initial temperature reading, and
t_f = final temperature reading.

Note A1.1—Typically 0.00076 for thermometers calibrated in °C and filled with a low hazard thermometric liquid.

Example:

A thermometer was immersed to 16°C; its initial reading, t_i, was 24.127°C; its final reading, t_f, was 27.876; the mean temperature of the emergent stem, T, was 26°C.

$$C_e = 0.00076 \times (27.9 − 24.1) \times (27.9 + 24.1 − 16 − 26) = 0.029 \quad (A1.6)$$

A1.1.3.2 *Thermometers Calibrated and Used in Partial Immersion, But at a Different Temperature Than the Calibrated Temperature:*

$$C_e = K(t_f − t_i)(t_o − t_c) \quad (A1.7)$$

where:

C_e = emergent stem correction,
K = differential coefficient of thermal expansion for the thermometric liquid, (See Note A1.2.)
t_i = initial temperature reading,
t_f = final temperature reading,
t_o = observed stem temperature, and
t_c = stem temperature at which the thermometer was calibrated.

Note A1.2—Typically 0.00076 for thermometers calibrated in °C and filled with a low hazard thermometric liquid.

Example:

A thermometer has an initial reading, t_i, 27°C; a final reading, t_f, 30°C; the observed stem temperature, t_o, 28°C; and the calibration temperature, t_c, 22°C.

$$C_e = 0.00076 \times (30 − 27) \times (28 − 22) = 0.014 \quad (A1.8)$$

A2. PRECISION STATISTICS

A2.1 The precision of this test method, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined for the following materials as listed in Table A2.1.

TABLE A2.1 Repeatability (S_r, r) and Reproducibility (S_R, R) Parameters for Coal Samples

Material	Average	S_r	S_R	r	R
91-2 lvb	14473.32	18.95	44.67	55.81	124.98
91-1 hvAb	14719.75	25.68	40.04	71.86	112.02
91-5 hvAb	12927.36	24.09	35.78	67.41	100.51
89-4 hvCb	11976.68	28.72	29.74	80.37	83.26
91-4 hvCb	11303.64	14.09	47.10	39.41	132.93
90-1 subB	9416.21	20.10	69.52	56.25	195.65
89-7 subA	11979.54	37.51	47.70	104.94	133.46
91-6 subA	11395.46	27.26	59.45	76.28	166.28
89-6 subC	9981.04	27.50	58.44	76.94	163.51
Lignite	9059.36	34.76	57.62	95.85	161.35

A2.2 The precision of this test method for coke samples, characterized by repeatability (S_r, r) and reproducibility (S_R, R) has been determined from the results in Research Report RR:D05–1025.[11] The results are summarized in Table A2.2. Values are in Btu/lb.

A2.3 *Repeatability Standard Deviation (S_r)*—The standard deviation of test results obtained under repeatability conditions. $r = 2.8 \times S_r$

Values are Btu/lb.

[11] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D05-1025. Contact ASTM Customer Service at service@astm.org.

TABLE A2.2 Repeatability (S_r, r) and Reproducibility (S_R, R)
Parameters for Coke Samples

Material	Type	Average	S_r	S_R	r	R
Reference 2	Furnace	13 062.63	13.45	32.07	37.67	89.79
CK9904	Furnace	13 047.00	24.62	40.68	68.96	113.90
CK0002	Breeze	12 984.75	20.98	41.65	58.75	116.62
CK0101	Furnace	13 070.88	15.52	42.66	43.46	119.44

A2.4 *Reproducibility Standard Deviation* (S_R)—The standard deviation of test results obtained under reproducibility conditions. R = 2.8 × S_X
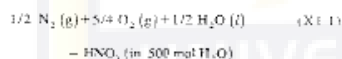
APPENDIXES

(Nonmandatory Information)

X1. THERMOCHEMICAL CORRECTIONS

X1.1 *Energy of Formation of Nitric Acid (HNO₃):*

X1.1.1 A correction, e1, (10.6.1) is applied for the formation of nitric acid.

X1.1.2 (1) HNO_3 is formed in the calorimeter by the following reaction:

$$1/2 \ N_2 \ (g) + 5/4 \ O_2 \ (g) + 1/2 \ H_2O \ (l) \qquad (X1.1)$$

$$\rightarrow HNO_3 \ (in \ 500 \ mol \ H_2O)$$

X1.1.3 The energy of formation of HNO_3 in approximately 500 mol of water under bomb conditions is minus 59.7 kJ/mol (14.3 kcal/mol). (7, 8) The enthalpy of reaction (all values in kJ/mol @ 298.15 K) is calculated as $\Delta_r H = \Delta_f H$ [HNO_3 (in 500 H_2O)] – ½ $\Delta_f H$ [N_2] – ¾ $\Delta_f H$ [O_2] – ½ $\Delta_f H$ [H_2O (l)] = -206.974 – ½ (0) – ¾ (0) – ½ (-285.830) = - 64.059. From this, the energy of reaction at constant volume is calculated from the following relationship: $\Delta_r U = \Delta_r H - RT\Delta n$ where R is the universal gas constant [8.314 J / (mol K)], T is the standard thermochemical reference temperature (298.15 K) and Δn is the change in the number of moles of gas in the reaction. RT – 2.479 kJ / mol. $\Delta_r U = -64.059 – (2.479) (-7/4) = -59.7$ kJ/mol.

X1.1.4 Normal convention assigns a negative value for a heat of formation that is exothermic. By definition, heat released from combustion processes are expressed as positive values. Hence, the negative factors developed for nitric and sulfuric acid corrections are expressed as positive values in the calculations.

X1.2 *Energy of Formation of Sulfuric Acid* (H_2SO_4)—By definition (see Terminology D121), the gross calorific value is obtained when the product of the combustion of sulfur in the sample is SO_2 (g). However, in actual bomb combustion processes, all the sulfur is found as H_2SO_4 in the bomb washings.

X1.2.1 A correction e3 is applied for the sulfur in the test sample that is converted to H_2SO_4. This correction is based upon the energy of formation of H_2SO_4 from SO_2 in solutions typically present in the bomb at the end of combustion. This correction is applied in one of two ways depending on whether

the nitric acid is calculated or the bomb washings are titrated. When a nominal 1 gram sample is burned, the resulting H_2SO_4 condensed with water formed in the bomb will have a ratio of about 15 mol of water to 1 mol of H_2SO_4. For this concentration, the energy of the reaction under the conditions of the bomb process is -297.2 kJ/mol. (8) As a result, a correction of 9.27 kJ (2.21 kcal) must be applied for each gram of sulfur converted to sulfuric acid. However, when the bomb washings are titrated, a correction of 2 × 59.7 kJ/mol (2 × 14.3 kcal/mol) of sulfur is already included in the total acid correction. Therefore the correction which must be applied for sulfur is 297.2 – (2 × 59.7) kJ/mol or 5.55 kJ/g of sulfur. This is expressed as 55.5 J (13.3 cal) for each percentage point of sulfur per gram of sample.

$$SO_2 + \frac{1}{2} \ O_2 \ (g) + H_2O \ (l) \rightarrow H_2SO_4 \ (in \ 15 \ mol \ H_2O) \quad (X1.2)$$
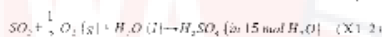
X1.2.2 The values above are based on a sample containing approximately 5 % sulfur and approximately 5 % hydrogen. The assumption is also made that the H_2SO_4 is dissolved entirely in the water condensed during combustion of the sample. (9) The enthalpy of reaction (all values in kJ/mol @ 298.15 K) is calculated as:

$$\Delta_r H = \Delta_f H \ [H_2SO_4 \ (in \ 15 \ H_2O)] - \Delta_f H \ [SO_2]$$

$$\frac{1}{2} \Delta_f H \ [O_2] - \Delta_f H \ [H_2O \ (l)]$$

$$= - 885.624 - (- 296.830) - \frac{1}{2} (0) - (- 285.830)$$

$$= - 300.963$$

From this, the energy of reaction at constant volume is calculated following the same procedure as in X1.1.3. $\Delta_r U = -300.963 – (2.479) (-3/2) = -297.2$ kJ/mol.

X1.2.3 For different sample weights or sulfur content, or both, the resultant normality of acid formed can be different, and therefore, the normality of titrant must be adjusted accordingly. Basing the calculation upon a sample of comparatively large sulfur content reduces the possible overall errors, because, for small percentages of sulfur, the correction is smaller.

X1.3 *Fuse Correction*—The energy required to melt a platinum or palladium wire is constant for each experiment if the same amount of platinum or palladium wire is used. As the energy is small, its effect is essentially cancelled out in the relationship between the standardization experiments and the calorific value determinations, and it can be neglected.

X1.4 *Reporting Results in Other Units:*

X1.4.1 Express the gross calorific value in joules per gram, calories per gram, or British thermal units per pound. The relationships between these units are given below.

1 International Table (IT) calorie	= 4.1868 J (10)
1 thermochemical calorie	= 4.184 J
1 IT cal/g	= 1.8 Btu/lb
1 Btu	= 1055.06 J
10^6 Btu or 1 MBtu	= 1.05506 GJ

Prior to widespread adoption of SI units, the unit MBtu was originally defined as one thousand Btu, presumably from the Roman numeral system where "M" stands for one thousand. Today, this is easily confused with the SI mega (M) prefix, which multiplies by a factor of one million. In an attempt at avoiding confusion, many still use MMBtu to represent one million Btu.

1 Btu/lb	= 2.326 J/g
1000 Btu/lb	= 2.326 GJ/t

X1.4.2 The combustion of fuel to produce energy results in the emission of combustion byproducts. The exact levels and types of each emission depend on the type of fuel used, the efficiency and age of the equipment, emissions controls that may be mandated by regulatory agencies, and other factors. An emissions factor is a value that attempts to relate the quantity of an emission released to the atmosphere with an activity associated with its release. For coal, these factors are often expressed as the mass of the emitted material divided by energy released during the combustion. Such factors facilitate estimation of emissions from various sources. Carbon dioxide (CO_2) emissions from the combustion of fuel can be determined accurately regardless of how the fuel is used as these emissions depend almost exclusively on the carbon content of the fuel. The same is true for sulfur dioxide (SO_2), where it originates from the sulfur content of the fuel. Both carbon and sulfur are almost completely oxidized during combustion and all carbon and sulfur in the fuel are assumed to be present in the flue gases as CO_2 and SO_2 respectively. The energy released may be expressed as either the net heat of combustion or lower heating value (LHV) or the gross heat of combustion or higher heating value (HHV). The net heat of combustion is generally closest to the actual energy yield. The gross heat of combustion which includes condensation of the combustion products is greater by about 5% in the case of coal and depends mainly on the hydrogen content of the fuel. The appropriateness of using LHV or HHV when calculating emission factors depends upon the application. For combustion where exhaust gases are cooled before discharging, HHV is more appropriate. Where no attempt is made to extract useful work from hot exhaust gases, the LHV is more suitable. In practice, many European publications report LHV, whereas North American publications use

HHV. General formulas to calculate emission factors for CO2 and SO2 are given below.

X1.4.2.1 To express carbon as kilograms of carbon dioxide per gigajoule:

$$CO_2, kg/GJ = (C, \% \times 44./12. / Heat\ of\ combustion,\ J/g) \times 10000$$

This is numerically equal to metric tons (t) of carbon dioxide per terajoule (TJ). The factor 44/12 is the ratio of the molecular weights of CO_2 and carbon. The aforementioned factor is used in routine commercial practice rather than the exact ratio of the molecular weights 44.0098/12.011. This results in an error of the reported value of no more than 0.1%.

In non-SI units:

$$CO_2, lb/MBtu = (C, \% \times 44./12. / Heat\ of\ combustion,\ Btu/lb) \times 10000$$

X1.4.2.2 To express sulfur as kilograms of sulfur dioxide per gigajoule:

$$SO_2, kg/GJ = (S, \% \times 64./32. / Heat\ of\ combustion,\ J/g) \times 10000$$

The factor 64./32. is the ratio of the molecular weights of SO_2 and sulfur.

In non-SI units:

$$SO_2, lb/MBtu = (S, \% \times 64./32. / Heat\ of\ combustion,\ Btu/lb) \times 10000$$

X1.5 *Sample Calculations:*

X1.5.1 *Heat Capacity:*

E	=	[(Hc × m) + e1 + e2] / t
Hc	=	26454 J/g;
m	=	1.0047g
$e1$	=	7.20 ml × 0.1000 N × 59.7 J = 43.0 J acid correction [by titration]
NAF	=	43.0 J / (26.454 kJ/g × 1.0047g) = 1.62 J/kJ of released energy
$e2$	=	K_f (Chromel C) × 57 mm wire consumed = 0.96 J/mm × 57 mm = 55 J, fuse correction
t	=	2.6006°C
E	=	[(26454 J/g × 1.0047 g) + 43.0 J + 55 J] / 2.6006°C, and
E	=	10258 J/°C.

X1.5.2 *Heat of Combustion [titration method]:*

Q_{ad} (gross)	=	[(t E) - e1 - e2 - e3 - e4] / m
E	=	10258 J/°C
t	=	2.4170°C
$e1$	=	12.90 ml × 0.1000 N × 59.7 J = 77.0 J, total acid correction
$e2$	=	K_f (Chromel C) × 54 mm wire consumed = 0.96 J/mm × 54 mm = 52 J, fuse correction
$e3$	=	55.5 × 1.24 % S × 0.7423 g = 51.1 J, sulfur correction
$e4$	=	46025 J/g × 0.2043 g = 9403 J, combustion aid correction
m	=	0.7423 g, mass of sample
Q_{ad}	=	[(10258 J/°C × 2.4170 °C) - 77.0 J - 52 J - 51.1 J - 9403 J] / 0.7423 g; and

Q_{vad} = 20491 J/g

X1.5.3 *Heat of Combustion [calculated nitric acid method]*:

Q_{vad} (gross) = [(t E) – e1 – e2 – e3 – e4] / m
E = 10258 J/°C
t = 2.4170°C
NAF = 1.62 J/kJ of released energy [from X1.5.1]
e1 = 1.62 / 1000 × 10258 J/°C × 2.4170°C = 40.2 J, nitric acid correction
e2 = K_t (Chromel C) × 54 mm wire consumed × 0.96 J/mm × 54 mm = 52 J, fuse correction

e3 = 92.7 × 1.24 % S × 0.7423 g = 85.3 J, sulfur correction
e4 = 46025 J/g × 0.2043 g = 9403 J, combustion aid correction
m = 0.7423 g, mass of sample
Q_{vad} = [(10258 J/°C × 2.4170 °C) – 40.2 J – 52 J – 85.3 J – 9403 J] / 0.7423 g, and
Q_{vad} = 20495 J/g

X2. OXIDATION OF COAL AFTER SAMPLING

X2.1 Gross Calorific Value Stability of Coal

X2.1.1 Table X2.1 illustrates the trend in the gross calorific value of twelve coal samples over a three and a half year time period. The results are dry basis values expressed in joules / gram. The proficiency test result for each sample represents the consensus value from 85 to 120 laboratories. The stability study was conducted in a single location on a single bottle of each material over the time frame listed in the table. With the exception of the anthracite sample, the heating values diminished anywhere from 250 to 950 J/g. The average monthly degradation is in the range of 6 to 23 J/g. The high end of this range is nine percent of the reproducibility limit of this Standard Test Method for bituminous coal.

X2.1.2 It is apparent from these results that the heating value of the majority of coals definitely decreases with time.

From the time it leaves the mine, coal starts to degrade. All coals, other than anthracites, tend to be very sensitive to oxidation. Exposure to air at ambient temperature causes deterioration to the extent that various properties are affected, including the heating value. Details of the oxidation can be explained in terms of chemical changes but the mechanisms of oxidation are unclear. If the heating value is changing as a result of these chemical changes then it is fair to assume that the amount of the fundamental contributing constituents to the heating value, carbon and hydrogen, must also be changing. As a result, it is ill advised to use coal samples as a check for bias or for routine quality control or quality assurance purposes without taking this degradation into account.

TABLE X2.1 Stability Monitoring of the Gross Calorific Value of Proficiency Test Samples

Sample Source	Canada Alberta	Columbia	South Africa	Canada British Columbia	Illinois USA IBC 105	NIST 2685b	China	Germany	Spain	Kentucky USA	Kentucky USA	Kentucky USA
Rank	SubC	hvAb	hvBb	mvb	hvCb	hvAb	an	mvb	hvCb	hvAb	hvAb	hvAb
Date	1999-1	1999-2	1999-3	1999-4	2000-1	2000-2	2000-3	2000-4	2001-1	2001-2	2001-3	2001-4
Consensus value	22243	27806	28420	32726	24993	32943	27485	33973	28866	31929	31326	31962
Initial value	22157	27734	28329	32777	24935	32915	27394	33973	28765	32019	31408	31945
Initial-Consensus value	-86	-73	-91	58	-57	-28	-92	1	-101	90	80	-17
3/Dec/01	21683	27312	28347	32715	24391	32787	27329	33844	28371	31824	31289	31991
3/Dec/01	21676	27282	28054	32618	24337	32737	27414	33789	28287	31758	31269	31958
6/Feb/02	21648	27277	28329	32682	24309	32739	27392	33749	28085	31705	31245	31888
6/Feb/02	21595	27268	28331	32667	24331	32759	27439	33707	28135	31790	31293	31896
8/Apr/02	21602	27295	28252	32604	24331	32779	27457	33805	28035	31633	31157	31782
8/Apr/02	21597	27286	28245	32648	34350	32717	27398	33790	28026	31702	31150	31804
14/Jun/02	21553	27235	28175	32566	24271	32714	27447	33727	27940	31621	31204	31882
14/Jun/02	21574	27172	28266	32597	24205	32674	27738	33754	27962	31650	31156	31892
10/Dec/02	21443	27155	28256	32754	24667	32054	27446	33694	27771	31584	31103	31751
10/Dec/02	21428	27155	28284	32678	24715	32640	27454	33756	27848	31655	31036	31771
24/Jun/04	21199	26844	28145	32508	24217	32546	27422	33701	27607	31423	30885	31547
24/Jun/04	21225	26873	28115	32465	24294	32476	...	...	27606	31381	30972	31586
20/Jun/05	21208	26847	28031	32425	24095	...	27480	33327	27359	31222	30824	31447
20/Jun/05	21248	26663	28868	32408	24140	...	27408	33475	27292	31229	30818	31498
Average Degradation	451	392	341	250	246	251	-73	420	973	565	458	492
Degradation/month [J/g]	10.6	9.2	8.0	5.9	5.8	9.2	-1.7	9.9	22.9	13.3	10.8	11.6

X3. TEMPERATURE DEPENDENCE OF THE HEAT OF COMBUSTION VALUE

X3.1 In an actual calorimetric determination, the calorimeter and its contents undergo an increase in temperature as a result of the bomb reaction. In order to obtain, from this observed temperature rise, the heat of combustion at some reference temperature that differs from the actual test temperature, it is necessary to have an understanding of the effective heat capacity of both the pre- and post-combustion systems. The difference in these quantities allows one to determine the temperature dependence of the heat of combustion value.

X3.2 For the majority of hydrocarbons, including coal, the temperature dependence of the heat of combustion value is dominated by the difference in the latent heat of vaporization of water, originating from the fuel derived hydrogen in the sample, between the test and reference temperatures. Each percentage point of fuel hydrogen, in a one gram sample, yields 90 mg of liquid water in the bomb. The temperature dependence of the latent heat of vaporization of water contributes directly to the temperature dependence of the resulting bomb determined gross heating value. For example, if a one gram (dry basis) combustion sample containing 5.56% fuel hydrogen is burned, the resulting amount of liquid water formed in the bomb is half a gram. In this case, the temperature dependence of the latent heat of vaporization results in a difference in the bomb determined gross heat of combustion of -14 J/g going from 25 to 35°C.

X3.3 Correcting the heat of combustion at constant volume determined at an arbitrary test temperature (t) to some other reference temperature, for example 25°C is as follows:

$$Qv(25) = Qv(t)\left[H - 0.09 + \left[\Delta u ... \Delta u_{(t)}(25)\right]\right] \quad (X3.1)$$

where:

Qv = the constant volume gross heat of combustion of the test sample.

H = the hydrogen content of the sample in weight percent excluding the hydrogen contributed by the sample moisture.

$\Delta u_{(t)}$ = the difference in the specific internal energy of water between the gaseous and liquid states. This is otherwise known as the latent heat of vaporization of water at constant volume.

Table X3.1 offers values of the specific internal energy of water over the temperature range of 20 to 40 °C. The pressure dependence of this value up to pressures typically associated with bomb calorimetric testing is insignificant. The change in the specific internal energy of water over any five degree increment in the range of 20 to 40 C is -14 J/g.

X3.4 While not imperceptible, the variation in the heating value over a modest temperature span is just a small fraction of the repeatability of the standard test method. The hydrogen content of benzoic acid (C_6H_5COOH) is 4.95%. The dry basis hydrogen content of the majority of coals is between 4 and 6%. When the test sample contains roughly the same amount of hydrogen as the calibrant, a practical approximation of the above relationship (X3.1) is realized by simply using the heat of combustion value of the calibrant at the reference temperature when standardizing a calorimeter that is operated at a different test temperature.

TABLE X3.1 Specific Internal Energy of Water[a]

Temperature / °C	u_g (gas) / Jg	u (liquid) / Jg	Δu / Jg
20	2402.8	83.83	2319
25	2409.8	104.74	2305
30	2416.9	125.66	2291
35	2423.9	146.57	2277
40	2430.9	167.48	2263

[a] L. Haar, J. G. Gallagher, and G. G. Kell, NBS/NRC Steam Tables, Hemisphere Publishing Corp., 1984.

REFERENCES

(1) ASTM Manual on Presentation of Data and Control Chart Analysis (MNL 7).

(2) Johnson, W.H., "The Enthalpies of Combustion and Formation of Acetanilide and Urea," J Res Nat Bur Std, 1975, 79a, 487.

(3) Laties, J., Rogster, B., Stener, N.J., "On the accuracy of rotating bomb combustion calorimetry for organic chlorine and bromine compounds," Chem Thermodynamics, 15 (6), 2, 683.

(4) Dickinson, H. C., Bulletin, U.S. Bureau of Standards, Vol. 11, 1915, p. 189.

(5) Prosen, J., Annalen der Physik Chemie, NFYA, Vol. 129, 1886, p. 315.

(6) Methods of Analysing & Testing, ... U.S. Bureau

... Bulletin 638, XMBUA, 1967, pp. 16-17.

(7) Mott, R. A., and Parker, C., "Studies in Bomb Calorimetry IV. Corrections," Fuel, FUELB, Vol. 34, 1955, p. 305-316.

(8) NBS/NBS Tables of Chemical Thermodynamic Properties: Selected values for Inorganic and C_1 and C_2 Organic Substances in SI Units, Journal of Phys. Chem. Ref. Data, 11, Supplement No. 2, 1982.

(9) Mott, R. A., and Parker, C., "Studies in Bomb Calorimetry IX. Formation of Sulfuric Acid," Fuel, Fuel B, Vol. 37, 1958, p. 11.

(10) Cohen, E. R., and Taylor, B. N., "The 1986 CODATA Recommended Values for the Fundamental Physical Constants," Journal of Phys. Ref. Data, Vol 17, No. 4, 1988, pp. 1795-1803.

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SUMMARY OF CHANGES

Committee D05 has identified the location of selected changes to this standard since the last issue (D5865-12) that may impact the use of this standard. (Approved Oct. 1, 2013.)

(1) Precision and Bias statement, Table 5 and Table 6 updated to reflect repeatability and reproducibility information for Coke.

(2) Table A2.1 and Table A2.2 added to Annex A2.
(3) Annex A2 revised to include data from RR:D05-1025
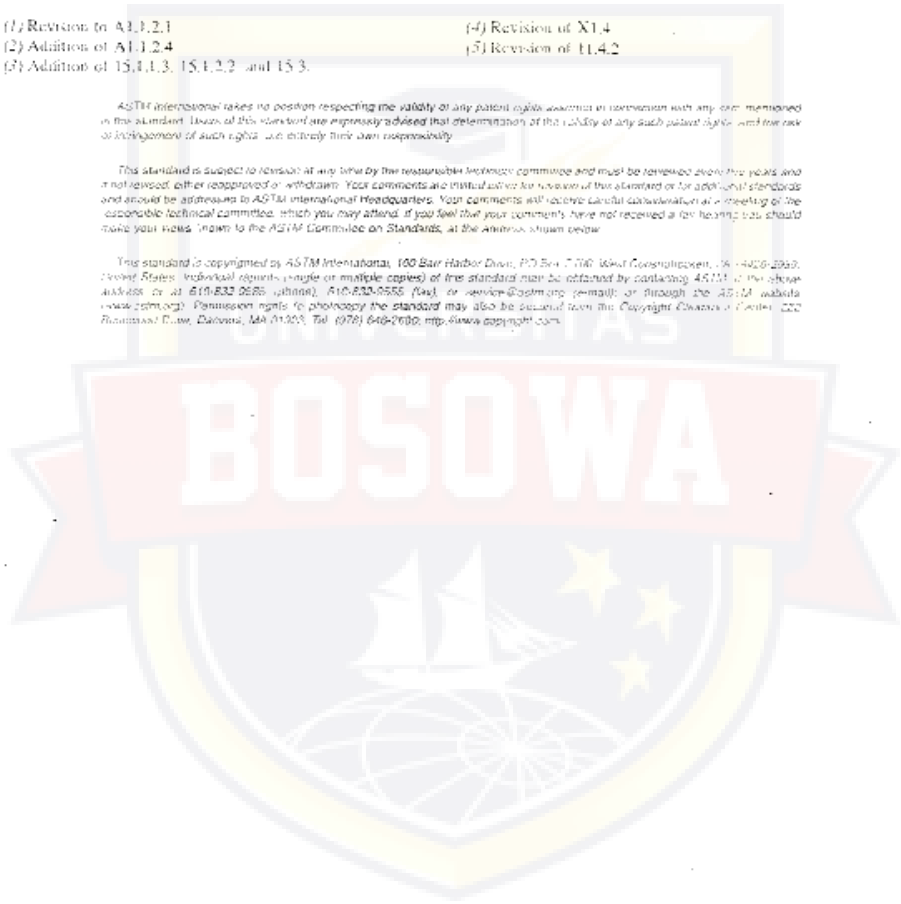(4) Section 12.6.3 revised for clarity

Committee D05 has identified the location of selected changes to this standard since the last issue (D5865-11a) that may impact the use of this standard. (Approved Sept. 1, 2012.)

(1) Revision to A1.1.2.1
(2) Addition of A1.1.2.4
(3) Addition of 15.1.1.3, 15.1.2.2 and 15.3.

(4) Revision of X1.4
(5) Revision of 11.4.2

Lembar Data Keselamatan Bahan

Lembar Data Keselamatan Bahan

Berdasarkan EC Directive 91/155/EEC

Tanggal terbit :	07.12.2005
Edisi pengganti dari	09.06.2005

1. Identifikasi bahan/preparasi dan negara/tempat mendapatkan

Identifikasi produk

No katalog :	106391
Nama produk :	Sodium carbonate decahydrate GR for analysis ISO,Reag. Ph Eur

Penggunaan bahan/preparasi

Reagen untuk analisa
Produksi bahan kimia

Identifikasi Perusahaan/Penanggung jawab

Perusahaan :	Merck KGaA * 64271 Darmstadt * Germany * Phone: +49 6151 72 0
No. Telefon darurat :	Customer Call Centre : + 62 0800 140 1253 (Toll Free)

2. Komposisi/informasi pada kandungan bahan

CAS No.:	6132 02 1	EC Index No.:	011 005 00 2
M :	286.14 g/mol	EC No.:	207 838 8
Formula Hill:	$CNa_2O_3 * 10 H_2O$		
Formula kimia :	$Na_2CO_3 * 10 H_2O$		

3. Identifikasi bahaya

Mengiritasi mata.

4. Pertolongan pertama

Setelah menghirup : hirup udara segar.
Setelah kontak dengan kulit: cuci dengan air yang banyak. Lepaskan pakaian yang terkontaminasi.
Setelah kontak dengan mata: bilas dengan air yang banyak dengan kelopak mata terbuka lebar.
Hubungi dokter mata.
Setelah menelan: segera beri korban air minum yang banyak. Hubungi dokter.

5. Tindakan pencegahan kebakaran

Media pemadam yang cocok :
Pada penyesuaian terhadap bahan yang ditempatkan di lingkungan terdekat.

Resiko khusus :
Tidak mudah menyala.

Peralatan pelindung khusus untuk kebakaran:
Jangan tinggal di zona berbahaya tanpa melengkapi diri dengan peralatan pernapasan.

Informasi lain :
Cegah air pemadam kebakaran memasuki air permukaan atau air tanah.

Lembar Data Keselamatan Merck

No katalog :	106391
Nama produk :	Sodium carbonate decahydrate GR for analysis ISO,Reag. Ph Eur

6. Tindakan terhadap tumpahan dan kebocoran

Tindakan pencegahan untuk personil terkait : Hindari penghisapan debu. Hindari kontak dengan bahan. Pastikan pasokan udara segar didalam ruangan tertutup.

Tindakan perlindungan lingkungan: Jangan biarkan memasuki sistem pembuangan kotoran.

Prosedur pembersihan / penyerapan: Ambil dalam keadaan kering. Teruskan ke pembuangan. Bersihkan area yang terkena. Hindari pembentukan debu.

7. Penanganan dan penyimpanan

Penanganan :

Tidak ada persyaratan lebih lanjut.

Penyimpanan :

Tertutup sangat rapat. Kering. Pada +15°C hingga +25°C.

Persyaratan untuk ruang penyimpanan dan wadah : Jangan gunakan wadah yang terbuat dari logam ringan.

8. Kontrol pemaparan/perlindungan personal

Peralatan pelindung perorangan:

Pakaian pelindung harus dipilih secara spesifik untuk tempat bekerja, tergantung konsentrasi dan jumlah bahan berbahaya yang ditangani. Daya tahan pakaian pelindung kimia harus dipastikan dari masing masing suplier.

Pelindung pernapasan : diperlukan ketika debu dihasilkan.

Pelindung mata : diperlukan

Pelindung tangan :

Dengan kontak penuh:
Bahan sarung tangan:	Karet nitrile
Ketebalan lapisan:	0.11 mm
Waktu terobosan:	> 480 Min.

Pada saat terkena percikan:
Bahan sarung tangan:	Karet nitrile
Ketebalan lapisan:	0.11 mm
Waktu terobosan:	> 480 Min.

Sarung tangan pelindung yang digunakan harus mengikuti spesifikasi pada EC directive 89/686/EEC dan standar gabungan d EN374, untuk contoh KCL 741 Dermatrill L (kontak penuh), 741 Dermatrill L (kontak percikan). Waktu terobosan yang disebutkan diatas ditentukan oleh KCL dalam uji laboratorium berdasarkan EN374 dengan sampel tipe sarung tangan yang dianjurkan.
Rekomendasi ini berlaku hanya untuk produk yang disebutkan dalam lembar data keselamatan dan diberikan oleh kami sesuai tujuan yang kami maksud. Ketika melarutkan dalam atau mencampurkan dengan bahan lain dan di bawah kondisi yang menyimpang dari kondisi dari yang disebutkan dalam EN374 silahkan hubungi suplier sarung tangan CE resmi (misal KCL GmbH, D 36124 Eichenzell, Internet: www.kcl.de).

Higiene indusri :
Segera ganti pakaian yang terkontaminasi. Gunakan krim pelindung kulit. Cuci tangan dan muka setelah bekerja dengan bahan tersebut.

Lembar Data Keselamatan Merck

Berdasarkan EC Directive 91/155/EEC

No katalog :	106391
Nama produk :	Sodium carbonate decahydrate GR for analysis ISO,Reag. Ph Eur

9. Sifat fisik dan kimia

Bentuk :		padat	
Warna :		putih	
Bau :		tidak berbau	
nilai pH			
pada 50 g/l H_2O	(25 °C)	11 12	
Titik lebur		33	°C
Titik didih		tidak tersedia	
Suhu penyalaan		tidak tersedia	
Titik nyala		tidak tersedia	
Batas ledakan	lebih rendah	tidak tersedia	
	lebih tinggi	tidak tersedia	
Densitas	(20 °C)	1.44	g/cm^3
Densitas bagian terbesar		~ 700 900	kg/m^3
Kelarutan dalam			
· air	(20 °C)	~ 210	g/l

10. Stabilitas dan reaktifitas

Kondisi yang harus dihindari

Pemanasan kuat.

Bahan yang harus dihindari

Reaksi yang hebat dapat terjadi dengan : aluminium, logam alkalin tanah dalam bentuk bubuk, senyawa nitro organik, fluorine, logam alkali, nonmetallic oxides / panas, konsentrasi sulfuric acid.

Produk penguraian yang berbahaya
tidak ada informasi yang tersedia

11. Informasi toksikologik

Toksisitas akut

LC_{50} (penghirupan, tikus): 2.3 mg/l /2 h (senyawa anhydrous) (IUCLID).
LD_{50} (oral, tikus): 4090 mg/kg (senyawa anhydrous) (IUCLID).

Gejala khusus pada hewan percobaan: Uji iritasi pada mata (kelinci): Iritasi (senyawa anhydrous) (IUCLID).
Uji iritasi pada kulit (kelinci): Iritasi ringan (senyawa anhydrous) (OECD 404).

Toksisitas subakut sampai kronik

Mutagenisitas bakterial : Escherichia coli: negatif. (senyawa anhydrous) (Lit.)
Tidak ada efek teratogeniik pada hewan percobaan. (senyawa anhydrous) (IUCLID)

Lembar Data Keselamatan Merck

Berdasarkan EC Directive 91/155/EEC

No katalog :	106391
Nama produk :	Sodium carbonate decahydrate GR for analysis ISO.Reag. Ph Eur

Informasi toksikologik lebih lanjut

Setelah menghirup debu: iritasi mukosa parah.
Setelah terjadi kontak dengan kulit: Iritasi ringan.
Setelah kontak mata : Iritasi.
Setelah tertelan : terjadi iritasi pada membran mukosa di mulut, pharynx, oesophagus dan saluran gastrointestinal.

Data lebih lanjut

Produk ini harus ditangani dengan hati hati lazimnya jika menangani bahan kimia.

12. Informasi ekologik

Degradasi biologik: Metoda untuk mengukur biodegradabilitas tidak dapat digunakan untuk bahan inorganik.

Perilaku dalam lingkup lingkungan: Konsentrasi dalam organisme tidak diharapkan.

Efek ekotoksik: Efek biologik:
Toksisitas pada ikan: L.macrochirus LC_{50}: 300 mg/l /96 h (senyawa anhydrous) (IUCLID);
Toksisitas daphnia : Daphnia magna EC_{50}: 265 mg/l /48 h (senyawa anhydrous) (IUCLID).

Data ekologik lebih lanjut: Diharapkan tidak ada masalah ekologi jika produk ditangani dan digunakan dengan hati hati dan penuh perhatian.

13. Pertimbangan pembuangan

Produk :

Bahan kimiä harus diatur sesuai dengan peraturan nasional masing masing. Pada www.retrologistik.de anda akan menemukan informasi spesifik mengenai negara dan bahan serta patner kontak.

Pengemasan :

Kemasan produk Merck harus dibuang sesuai peraturan spesifik negara atau harus melewati sistem pengembalian kemasan (packaging return system). Pada www.retrologistik.de anda akan menemukan informasi khusus mengenai kondisi nasional masing masing serta kontak patner.

14. Informasi transport

Tidak ada subjek peraturan transport.

15. Informasi peraturan

Pelabelan menurut EC Directive

Simbol :	Xi	Mengiritasi
R phrases:	36	Mengiritasi mata.
S phrases:	22 26	Jangan menghirup debu. Dalam kasus terjadi kontak dengan mata, bilas langsung dengan air yang banyak dan minta nasihat medik.
EC No.:	207 838 8	EC label

16. Informasi lain:

Lembar Data Keselamatan Merck

Berdasarkan EC Directive 91/155/EEC

No katalog :	106391
Nama produk :	Sodium carbonate decahydrate GR for analysis ISO,Reag. Ph Eur

Alasan perubahan

Bab 7 : kondisi penyimpanan.

Perbaikan secara keseluruhan.

Perwakilan regional:

Alamat Merck Indonesia
Kantor Pusat: Jl T.B Simatupang No 8 Pasar Rebo Jakarta 13760 * Phone: +62 21 8400081, +62 21 87791415
Kantor Marketing: Gedung PP Plaza Jl. T.B Simatupang No 57 Jakarta 13760 * Phone: +62 21 8413889 *
email: Chemicals@merck.co.id

*Informasi yang terkandung di dalam ini berdasarkan pada pengetahuan terkini. Informasi ini
menggambarkan produk sesuai dengan tindakan pencegahan-dan keselamatan. Informasi ini tidak
menjamin sifat dari produk.*

Lembar Data Keselamatan Bahan

Menurut EC Directive 91/155/EEC

Tanggal terbit :　　　　　　　　　08.04.2004

1.　Identifikasi bahan/preparasi dan negara/tempat mendapatkan

Identifikasi produk

No katalog :　　　　101969

Nama produk :　　　　Silica gel with moisture indicator (orange gel) desiccant – 1   3 mm

Penggunaan bahan/preparasi

　　　　Reagen untuk analisa
　　　　Produksi bahan kimia

Identifikasi Perusahaan/tempat mendapatkan

Perusahaan :　　　　Merck KGaA * 64271 Darmstadt * Germany * Phone: +49 6151 72 0

No. Telefon darurat :　　　　Customer Call Centre : + 62 0800 140 1253 (Toll Free)

2.　Komposisi/informasi bahan

preparasi silica gel

3.　Identifikasi bahaya

Tidak ada produk yang berbahaya seperti dispesifikasi dalam Directive 67/548/EEC.

4.　Pertolongan pertama

Setelah terhirup : hirup udara segar
Setelah kontak dengan kulit : cuci dengan air yang cukup
Setelah kontak dengan mata : bilas dengan air
Setelah tertelan (dalam jumlah besar) : konsultasikan dengan dokter jika merasa tak enak

---

5.　Tindakan Pencegahan Kebakaran

Media pemadam yang cocok :
Dalam penyesuaian bahan, tempatkan di lingkungan

Resiko khusus :
Tidak mudah menyala mungkin melepaskan uap yang berbahaya.

Peralatan pelindung khusus untuk kebakaran:
Jangan tinggal di zona berbahaya tanpa menggunakan peralatan pernapasan

Informasi lain :
Cegah air pemdaman api memasuki air permukaan atau air tanah.

Lembar Data Keselamatan Bahan Merck

Menurut EC Directive 91/155/EEC

No katalog :   101969
Nama produk :   Silica gel with moisture indicator (orange gel) desiccant ~ 1 3 mm

6. Tindakan terhadap tumpahan dan kebocoran

Tindakan pencegahan untuk personil terkait : Hindari pembentukan debu; jangan menghirup debu.

Pengukuran proteksi bagi lingkungan: Jangan biarkan memasuki sistem pembuangan kotoran

prosedur pembersihan / penyerapan Keringkan. Teruskan ke pembuangan, bersihkan area yang terkena

7. Penanganan dan penyimpanan

Penanganan :

Tidak ada persyaratan lebih lanjut.

Penyimpanan :

Tertutup sangat rapat. Kering. Suhu penyimpanan : tidak ada batasan

8. Kontrol pemaparan/perlindungan personal

Peralatan pelindung perorangan

Pakaian pelindung harus dipilih sesuai tempat bekerja, tergantung konsentrasi dan jumlah bahan
berbahaya yang ditangani. Kekebalan pakaian pelindung kimia harus dipastikan dari suplier yang
baik

Pelindung pernapasan :   diperlukan ketika debu dihasilkan.

Pelindung tangan :   Dengan kontak penuh :
                                Bahan sarung tangan: Karet nitrile
                                Ketebalan lapisan   0.11   mm
                                Waktu terobosan: > 480 Min.

                                Pada saat terkena percikan:
                                Bahan sarung tangan: Karet nitrile
                                Ketebalan lapisan   0.40   mm
                                Waktu terobosan: > 480 Min.

                                Sarung tangan pelindung yang digunakan harus mengikuti spesifikasi pada
                                EC directive 89/686/EEC dan standar gabungan d EN374, untuk contoh
                                KCL 740 Dermatril® (kontak penuh), 730 Camatril® Velours (kontak
                                percikan). Pemecahan yang disebutkan diatas ditentukan oleh KCL dalam
                                uji laboratorium menurut EN374 dengan sampel tipe sarung tangan yang
                                dianjurkan.
                                Anjuran ini digunakan hanya untuk produk yang disebutkan dalam daftar
                                data keamanan dan disuplai oleh kami sepertidalam spesifikasi yang
                                kami maksudkan. Ketika dalam pelarutan atau pencampuran dengan bahan
                                lain   terdapat penyimpangan kondisi dari yang disebutkan dalam EN374
                                silahkan kontak suplier CE approved gloves (e.g. KCL GmbH, D 36124
                                Eichenzell, Internet: www.kcl.de).

Higiene industri :
Ganti pakaian yang terkontaminasi. Penggunaan cream pelindung kulit dianjurkan. Cuci tangan setelah
bekerja dengan bahan tersebut

Lembar Data Keselamatan Bahan Merck

Menurut EC Directive 91/155/EEC

No katalog :   101969
Nama produk :   Silica gel with moisture indicator (orange gel) desiccant – 1   3 mm

9. Sifat fisik dan kimia

Bentuk :	granul		
Warna :	orange		
Bau :	tak berbau		
nilai pH		tidak ada	
Titik lebur		> 1000	°C
Titik didih		tidak ada	
Suhu sumber nyala		tidak ada	
Titik nyala		tidak ada	
Batas ledakan	lebih rendah	tidak ada	
	lebih tinggi	tidak ada	
Berat jenis		tidak ada	
Berat jenis bulk		~ 800	kg/m^3
Kelarutan dalam air		tidak ada	
Penyusutan termal		823	°C

10. Stabilitas dan reaktifitas

Kondisi yang harus dihindari

Pemanasan kuat.

Bahan yang harus dihindari

tidak ada informasi yang tersedia

Produk menyusut yang berbahaya

tidak ada informasi yang tersedia

Informasi lebih lanjut

higroskopis.

11. Informasi toksikologik

Toksisitas akut

Data kuantitatif toksisitas produk ini tidak ada

Informasi toksikologik lebih lanjut

Sifat yang berbahaya relatif tidak mungkin terjadi.
Terhirup debu harus dihindari karena debu inert dapat merusak fungsi organ pernapasan

Data lebih lanjut

Produk ini harus ditangani dengan hati hati seperti terhadap bahan kimianya

Lembar Data Keselamatan Bahan Merck

Menurut EC Directive 91/155/EEC

No katalog :	101969
Nama produk :	Silica gel with moisture indicator (orange gel) desiccant ~ 1 - 3 mm

12. Informasi ekologik

Efek ekotoksik: Data kuantitatif pada efek ekologik produk ini tidak tersedia

Data ekologik lebih jauh : Diharapkan Tidak ada masalah ekologik ketika produk ditangani dan digunakan dengan hati hati dan penuh perhatian

13. Pertimbangan pembuangan

Produk :

Bahan kimia harus diatur, untuk menyesuaikan dengan peraturan nasional. Pada www.retrologistik.de anda akan menemukan negara dan bahan informasi yang spesifik sebaik kontak patner

Pengepakan :

Pengepakan produk Merck harus diatur untuk memenuhi peraturan spesifik negara atau harus melewati sistem pengembalian pengepakan (packaging return system). Pada www.retrologistik.de anda akan menemukan informasi khusus pada kondisi nasional yang diharapkan sebaik kontak patner

14. Informasi transport

Tidak ada subjek peraturan transport.

15. Informasi peraturan

Pelabelan menurut EC Directive

Simbol :

R phrases:

S phrases:

...

16. Informasi lain:

...

Perwakilan regional:

Alamat Merck Indonesia
Kantor Pusat: Jl T.B Simatupang No 8 Pasar Rebo Jakarta 13760 * Phone: +62 21 8400081, +62 21 87791415
Kantor Marketing: Gedung PP Plaza Jl. T.B Simatupang No 57 Jakarta 13760 * Phone: +62 21 8413889 *
email: Chemicals merck.co.id

Informasi yang terkandung didalam ini berdasarkan pengetahuan terbaru. Informasi ini menggambarkan produk sesuai dengan tindakan pencegahan dan keselamatan. Informasi ini tidak menjamin sifat dari produk.

MERCK

Lembar Data Keselamatan Bahan

Menurut EC Directive 91/155/EEC

Tanggal terbit :   16.04.2004

---

1. Identifikasi bahan/preparasi dan negara/tempat mendapatkan

Identifikasi produk

No katalog :   104086

Nama produk :   Glass wool LAB

Penggunaan bahan/preparasi

Produksi bahan kimia

Identifikasi Perusahaan/tempat mendapatkan

Perusahaan :   Merck KGaA * 64271 Darmstadt * Germany * Phone: +49 6151 72 0

No. Telefon darurat :   Customer Call Centre : + 62 0800 140 1253 (Toll Free)

---

2. Komposisi/informasi bahan

CAS No.:   65997 17 3

German Foodstuffs and   266 046 0
Consumer Goods Act

---

3. Identifikasi bahaya

Tidak ada produk yang berbahaya seperti dispesifikasi dalam Directive 67/548/EEC.

---

4. Pertolongan pertama

Setelah terhirup : hirup udara segar Konsultasikan dengan dokter bila ada keluhan
Setelah kontak pada kulit: cuci dengan air yang cukup. Lepaskan pakaian yang terkontaminasi
Setelah kontak dengan mata : bilas dengan air Panggil ophtamologis
Setelah tertelan : Berikan korban minum air yang cukup Konsultasikan dengan dokter bila ada keluhan

---

5. Tindakan Pencegahan Kebakaran

Media pemadam yang cocok :
Dalam penyesuaian bahan, tempatkan di lingkungan

Resiko khusus :
Tidak mudah menyala

---

6. Tindakan terhadap tumpahan dan kebocoran

Tindakan pencegahan untuk personil terkait : Hindari terjadi dan terhisap debuChemizorb®)

prosedur pembersihan / penyerapan Keringkan. Teruskan ke pembuangan, bersihkan area yang terkena,
hindari terjadinya debu

Lembar Data Keselamatan Bahan Merck

Menurut EC Directive 91/155/EEC

No katalog : 104086
Nama produk : Glass wool LAB

---

7. Penanganan dan penyimpanan

Penanganan :

Tidak ada persyaratan lebih lanjut.

Penyimpanan :

Tertutup sangat rapat. Kering. Suhu penyimpanan : tidak ada batasan

---

8. Kontrol pemaparan/perlindungan personal

Peralatan pelindung perorangan

Pakaian pelindung harus dipilih sesuai tempat bekerja, tergantung konsentrasi dan jumlah bahan berbahaya yang ditangani. Kekebalan pakaian pelindung kimia harus dipastikan dari suplier yang baik.

Pelindung pernapasan :   diperlukan ketika debu dihasilkan.

Pelindung mata :   dibutuhkan

Pelindung tangan :   Dengan kontak penuh:

Bahan sarung tangan:	Karet nitrile
Ketebalan lapisan:	0.11 mm
Waktu terobosan:	> 480 Min.

Pada saat terkena percikan:

Bahan sarung tangan:	Karet nitrile
Ketebalan lapisan:	0.11 mm
Waktu terobosan:	> 480 Min.

Sarung tangan pelindung yang digunakan harus mengikuti spesifikasi pada EC directive 89/686/EEC dan standar gabungan d EN374, untuk contoh KCL 740 Dermatril® (kontak penuh), 740 Dermatril® (kontak percikan). Pemecahan yang disebutkan diatas ditentukan oleh KCL dalam uji laboratorium menurut EN374 dengan sampel tipe sarung tangan yang dianjurkan.
Anjuran ini digunakan hanya untuk produk yang disebutkan dalam daftar data keamanan dan disuplai oleh kamu sepertidalam spesifikasi yang kami maksudkan. Ketika dalam pelarutan atau pencampuran dengan bahan lain terdapat penyimpangan kondisi dari yang disebutkan dalam EN374 silahkan kontak suplier CE approved gloves (e.g. KCL GmbH, D 36124 Eichenzell, Internet: www.kcl.de).

Higiene industri :
Ganti pakaian yang terkontaminasi . Cuci tangan setelah bekerja dengan bahan tersebut

---

9. Sifat fisik dan kimia

Bentuk :	padat
Warna :	tak berwarna hingga putih
Bau :	tak berbau
nilai pH	tidak ada
Titik lebur	tidak ada
Titik didih	tidak ada
Suhu sumber nyala	tidak ada
Titik nyala	tidak ada

Lembar Data Keselamatan Bahan Merck

Menurut EC Directive 91/155/EEC

| No katalog : | 104086 |
| Nama produk : | Glass wool LAB |

Batas ledakan	lebih rendah	tidak ada
	lebih tinggi	tidak ada
Berat jenis		tidak ada
Kelarutan dalam air	(20 °C)	tidak larut

10. Stabilitas dan reaktifitas

Kondisi yang harus dihindari

tidak ada informasi yang tersedia

Bahan yang harus dihindari

tidak ada informasi yang tersedia

Produk menyusut yang berbahaya

tidak ada informasi yang tersedia

11. Informasi toksikologik

Toksisitas akut

Data kuantitatif toksisitas produk ini tidak ada

Informasi toksikologik lebih lanjut

Setelah terhisap (partikel, debu) : Gejala iritasi pada saluran pernapasan
Penyerapan dalam jumlah besar dapat menyebabkan kerusakan berikut : paru paru.
Setelah kontak dengan kulit : iritasi ringan.
Setelah kontak mata ; iritasi ringan.

Data lebih lanjut

Produk ini harus ditangani dengan hati hati seperti terhadap bahan kimianya

12. Informasi ekologik

Efek ekotoksik: Data kuantitatif pada efek ekologik produk ini tidak tersedia

Data ekologik lebih janh : Diharapkan Tidak ada masalah ekologik ketika produk ditangani dan digunakan dengan hati hati dan penuh perhatian

13. Pertimbangan pembuangan

Produk :

Bahan kimia harus diatur, untuk menyesuaikan dengan peraturan nasional. Pada www.retrologisiik.de anda akan menemukan negara dan bahan informasi yang spesifik sebaik kontak patner

Pengepakan :

Pengepakan produk Merck harus diatur untuk memenuhi peraturan spesifik negara atau harus melewati sistem pengembelian pengepakan (packaging return system). Pada www.retrologistik.de anda akan menemukan informasi khusus pada kondisi nasional yang diharapkan sebaik kontak patner

Lembar Data Keselamatan Bahan Merck

Menurut EC Directive 91/155/EEC

No katalog :   104086
Nama produk :   Glass wool LAB

14. Informasi transport

Tidak ada subjek peraturan transport.

15. Informasi peraturan

Pelabelan menurut EC Directive

Simbol :

R phrases:

S phrases:

16. Informasi lain:

Perwakilan regional:

Alamat Merck Indonesia
Kantor Pusat: Jl T.B Simatupang No 8 Pasar Rebo Jakarta 13760 * Phone: +62 21 8400081, +62 21 87791415
Kantor Marketing: Gedung PP Plaza Jl. T.B Simatupang No 57 Jakarta 13760 * Phone: +62 21 8413889 *
email: Chemicals merck.co.id

*Informasi yang terkandung didalam ini berdasarkan pengetahuan terbaru. Informasi ini
menggambarkan produk sesuai dengan tindakan pencegahan dan keselamatan. Informasi ini tidak
menjamin sifat dari produk.*

MERCK

Lembar Data Keselamatan Bahan

Berdasarkan EC Directive 91/155/EEC

Tanggal terbit :	28.02.2005
Edisi pengganti dari	14.07.2004

1. Identifikasi bahan/preparasi dan negara/tempat mendapatkan

Identifikasi produk

No katalog : 105873

Nama produk : Magnesium perchlorate hydrate [about 83% Mg(ClO$_4$)$_2$], desiccant, about 1-4 mm

Penggunaan bahan/preparasi

Reagen untuk analisa

Identifikasi Perusahaan/Penanggung jawab

Perusahaan : Merck KGaA * 64271 Darmstadt * Germany * Phone: +49 6151 72 0

No. Telefon darurat : Customer Call Centre : + 62 0800 140 1253 (Toll Free)

2. Komposisi/informasi pada kandungan bahan

CAS No.:	64010 42 0		
Formula Hill;	Cl$_2$MgO$_8$ * x H$_2$O	EC No.:	233 108 3
Formula kimia :	Mg(ClO$_4$)$_2$ * x H$_2$O		

3. Identifikasi bahaya

Kontak dengan bahan yang mudah terbakar dapat mengakibatkan kebakaran. Mengiritasi mata, sistem pernapasan dan kulit.

4. Pertolongan pertama

Setelah menghirup : hirup udara segar. Hubungi dokter.
Setelah kontak pada kulit: cuci dengan air yang banyak. Lepaskan pakaian yang terkontaminasi. Hubungi dokter.
Setelah kontak dengan mata : bilas dengan air yang banyak dengan kelopak mata terbuka lebar. Hubungi dokter mata.
Setelah menelan: segera beri korban air minum yang banyak. Hubungi dokter.

Lembar Data Keselamatan Bahan Merck

Berdasarkan EC Directive 91/155/EEC

No katalog :	105873
Nama produk :	Magnesium perchlorate hydrate [about 83% Mg(ClO$_4$)$_2$], desiccant, about 1-4 mm

---

5. Tindakan pencegahan kebakaran

Media pemadam yang cocok :
Pada penyesuaian terhadap bahan yang ditempatkan di lingkungan terdekat

Resiko khusus :
Tidak mudah menyala. Penyulut api. Jauhkan dari bahan yang mudah menyala. Perkembangan gas atau uap menyala yang berbahaya mungkin terjadi dalam kejadian kebakaran. Berikut ini dapat berkembang pada saat kebakaran : hydrochloric acid.

Peralatan pelindung khusus untuk kebakaran:
Jangan berada di zona berbahaya tanpa peralatan pelindung pernapasan. Untuk menghindari kontak dengan kulit, jaga jarak aman dan gunakan pakaian pelindung yang sesuai.

Informasi lain :
Mengandung uap yang keluar dengan air. Cegah air pemadam kebakaran memasuki air permukaan atau air tanah.

---

6. Tindakan terhadap tumpahan dan kebocoran

Tindakan pencegahan untuk personil terkait : Hindari penghisapan debu. Hindari kontak dengan bahan. Pastikan pasokan udara segar didalam ruangan tertutup.

Tindakan perlindungan lingkungan: Jangan biarkan memasuki sistem pembuangan kotoran.

Prosedur pembersihan / penyerapan: Ambil dalam keadaan kering. Teruskan ke pembuangan. Bersihkan area yang terkena. Hindari pembentukan debu.

---

7. Penanganan dan penyimpanan

Penanganan :

Tidak ada persyaratan lebih lanjut.

Penyimpanan :

Tertutup sangat rapat. Secara terpisah atau bersama sama dengan bahan pengoksidasi lain saja dan jauhkan dari sumber nyala dan panas. Kering. Suhu penyimpanan : tidak ada batasan.

---

8. Kontrol pemaparan/perlindungan personal

Peralatan pelindung perorangan:

Pakaian pelindung harus dipilih secara spesifik untuk tempat bekerja, tergantung konsentrasi dan jumlah bahan berbahaya yang ditangani. Daya tahan pakaian pelindung kimia harus dipastikan dari masing-masing suplier.

Pelindung pernapasan :   diperlukan ketika debu dihasilkan. Filter P 3 (menurut DIN 3181) untuk partikel padat dan cair bahan toksik dan sangat toksik

Lembar Data Keselamatan Bahan Merck

Berdasarkan EC Directive 91/155/EEC

No katalog :	105873
Nama produk :	Magnesium perchlorate hydrate [about 83% $Mg(ClO_4)_2$], desiccant, about 1-4 mm

Pelindung tangan :	Dengan kontak penuh :	
	Bahan sarung tangan:	Karet nitrile
	Ketebalan lapisan:	0.11 mm
	Waktu terobosan:	> 480 Min.
	Pada saat terkena percikan:	
	Bahan sarung tangan:	Karet nitrile
	Ketebalan lapisan:	0.11 mm
	Waktu terobosan:	> 480 Min.

Sarung tangan pelindung yang digunakan harus mengikuti spesifikasi pada EC directive 89/686/EEC dan standar gabungan d EN374, untuk contoh KCL 741 Dermatril® L (kontak penuh), 741 Dermatril® L (kontak percikan). Waktu terobosan yang disebutkan diatas ditentukan oleh KCL dalam uji laboratorium berdasarkan EN374 dengan sampel tipe sarung tangan yang dianjurkan.

Rekomendasi ini berlaku hanya untuk produk yang disebutkan dalam lembar data keselamatan dan diberikan oleh kami sesuai tujuan yang kami maksud. Ketika melarutkan dalam atau mencampurkan dengan bahan lain dan di bawah kondisi yang menyimpang dari kondisi dari yang disebutkan dalam EN374 silahkan hubungi suplier sarung tangan CE resmi (misal KCL GmbH, D 36124 Eichenzell, Internet. www.kcl.de).

Higiene indusri :
Segera ganti pakaian yang terkontaminasi. Gunakan cream pelindung kulit. Cuci tangan dan muka setelah bekerja dengan bahan tersebut.

9. Sifat fisik dan kimia

Bentuk :		padat		
Warna :		putih		
Bau :		tidak berbau		
nilai pH				
pada 50 g/l H_2O	(25 °C)	~ 8.2		
Titik lebur		251	°C	terurai
Titik didih		tidak tersedia		
Suhu penyalaan		tidak tersedia		
Titik nyala		tidak tersedia		
Batas ledakan	lebih rendah	tidak tersedia		
	lebih tinggi	tidak tersedia		
Densitas	(25 °C)	2.60	g/cm³	(dihitung berdasarkan bahan murni)
Densitas bagian terbesar		~ 600-800	kg/m³	
Kelarutan dalam air	(25 °C)	~ 500	g/l	

Lembar Data Keselamatan Bahan Merck

Berdasarkan EC Directive 91/155/EEC

| No katalog : | 105873 |
| Nama produk : | Magnesium perchlorate hydrate [about 83% Mg(ClO$_4$)$_2$], desiccant, about 1-4 mm |

10. Stabilitas dan reaktifitas

Kondisi yang harus dihindari

tidak ada informasi yang tersedia

Bahan yang harus dihindari

Beresiko meledak dengan: bahan mudah terbakar, bahan yang dapat teroksidasi, alkenes, ammonia, dimethyl sulfoxide / panas, etanol, ethylene oxide, hydrocarbons / panas, mineral acids, pelarut organik, bahan organik, phosphorus, asam, logam dalam bentuk bubuk (Membentuk senyawa yang dapat meledak)

Produk penguraian yang berbahaya

Pada saat kebakaran. Lihat bab 5.

Informasi lebih lanjut

bahan penyulut api, higroskopis!

11. Informasi toksikologik

Toksisitas akut

Data kuanutatif toksisitas produk ini tidak tersedia.

Informasi toksikologik lebih lanjut

Setelah terhirup : Iritasi terjadi pada membran mukosa, batuk dan dyspnoea. Setelah kontak dengan kulit : Iritasi.
Setelah kontak dengan mata : Iritasi.
Setelah tertelan : terjadi iritasi pada membran mukosa di mulut, pharynx, oesophagus dan saluran gastrointestinal.
Efek sistemik : Gangguan CNS.

Data lebih lanjut

Sifat bahaya lebih lanjut tidak bisa dikesampingkan.
Produk ini harus ditangani dengan hati-hati lazimnya jika menangani bahan kimia.

12. Informasi ekologik

Efek ekotoksik: Data kuanutatif pada efek ekologi dari produk ini tidak tersedia.

Data ekologik lebih lanjut: Berikut berlaku untuk perchlorates secara umum :
Efek biologik :
Ikan : Leuciscus idus LC$_{50}$: 1850 mg/l/48 jam.
Daphnia: Daphnia magna EC$_{50}$: 1077 mg/l/24 jam.
Algae: Scenedesmus quadricauda IC$_{50}$: 360 mg/l/7 hari.
Bacteria: Microcystix aeruginosa EC$_{50}$: 79 mg/l/8 hari.

Efek ekologik yang merugikan tidak dapat diabaikan jika penanganan atau pembuangannya tidak tepat.

Jangan biarkan memasuki perairan, air limbah atau tanah!

Lembar Data Keselamatan Bahan Merck

Berdasarkan EC Directive 91/155/EEC

No katalog :	105873
Nama produk :	Magnesium perchlorate hydrate [about 83% Mg(ClO$_4$)$_2$], desiccant, about 1 4 mm

13. Pertimbangan pembuangan

Produk :

Bahan kimia harus diatur sesuai dengan peraturan nasional masing masing. Pada www.retrologistik.de anda akan menemukan informasi spesifik mengenai negara dan bahan serta patner kontak.

Pengemasan :

Kemasan produk Merck harus dibuang sesuai peraturan spesifik negara atau harus melewati sistem pengembalian kemasan (packaging return system). Pada www.retrologistik.de anda akan menemukan informasi khusus mengenai kondisi nasional masing masing serta kontak patner.

14. Informasi transport

Jalan & Rel ADR, RID
   UN 1475 MAGNESIUMPERCHLORAT, 5.1, II

Jalur perairan pedalaman (sungai) ADN, ADNR tidak diuji

Laut IMDG Code
   UN 1475 MAGNESIUM PERCHLORATE, 5.1, II
   Ems F H S Q

Udara CAO, PAX
   UN 1475 MAGNESIUM PERCHLORATE, 5.1, II

Peraturan pengangkutan (transportasi) dirujuk berdasarkan peraturan internasional dan dalam aturan yang berlaku di Jerman. Penyimpangan yang mungkin terjadi di negara lain tidak dipertimbangkan.

15. Informasi peraturan

Pelabelan menurut EC Directive

Simbol :	O	Mengoksidasi
	Xi	Mengiritasi
R phrases:	8 36/37/38	Kontak dengan bahan yang mudah terbakar dapat mengakibatkan kebakaran. Mengiritasi mata, sistem pernapasan dan kulit.
S phrases:	17 26	Jauhkan dari bahan yang mudah terbakar. Dalam kasus terjadi kontak dengan mata, bilas langsung dengan air yang banyak dan minta nasihat medik.

16. Informasi lain:

Alasan perubahan

Bab 15 : pelabelan.
Bab 8 : peralatan pelindung perorangan.
Bab 10 : stabilitas dan reakuifitas.

Perbaikan secara keseluruhan.

Perwakilan regional:

Alamat Merck Indonesia
Kantor Pusat: Jl T.B Simatupang No 8 Pasar Rebo Jakarta 13760 * Phone: +62 21 8400081, +62 21 87791415
Kantor Marketing: Gedung PP Plaza Jl, T,B Simatupang No 57 Jakarta 13760 * Phone: +62 21 8413889 *
email: Chemicals@merck.co.id

Lembar Data Keselamatan Bahan Merck

Berdasarkan EC Directive 91/155/EEC

No katalog :   105873
Nama produk :   Magnesium perchlorate hydrate [about 83% Mg(ClO$_4$)$_2$], desiccant, about 1-4 mm

Informasi yang terkandung di dalam ini berdasarkan pada pengetahuan terkini. Informasi ini menggambarkan produk sesuai dengan tindakan pencegahan dan keselamatan. Informasi ini tidak menjamin sifat dari produk.

PT. SURYA BIRU MURNI ACETYLENE
Industri Gas Acetylene, Oxygen & Nitrogen

LEMBAR DATA MATERIAL untuk KESELAMATAN (MSDS)

Karbon Dioksida, gas	MSDS No. 108

SPESIFIKASI TEKNIS

SPESIFIKASI

Karbon Dioksida	>99,8%
Klasifikasi gas	Tidak terbakar
Kelas & sub resiko	2.2
UN No.	1013

DATA FISIK

Simbol Kimia	CO_2
Titik sublimasi pada 1 atm.	- 78,5 °C
Berat Jenis Relatif (Udara = 1)	1,522
Berat Molekul	44,01
Suhu Kritis	31,1 °C
Tekanan Kritis	7382 kPa abs
Densitas Kritis	468 kg/m³
Berat Jenis Gas (@ 101,3 kPa & 21°C)	1,522 kg/m³
Daya Larut dalam air (@ 101,3 kPa & 20°C)	0,90 cm³/cm³
Isi Spesifik (@ 101,3 kPa & 21°C)	0,5457 m³/kg
Titik Bakar	tidak terbakar

URAIAN & SIFAT-SIFAT

Karbon dioksida adalah campuran dari 27,3 % Karbon dan 72,7 % Oksigen (satuan berat).

Karbon dioksida adalah gas yang tidak mudah bereaksi dan tidak beracun, tidak berwarna, tidak berbau, dan tidak membantu pembakaran maupun kehidupan manusia.

Gas karbon dioksida dikemas dalam bejana bertekanan, dengan berbagai ukuran (isi) dan satuan yang digunakan adalah berat.

PENGGUNAAN

- Industri makanan dan minuman.
- Industri kimia, misalnya membuat Sodium karbonat.
- Untuk proses pengelasan logam (MIG welding)
- Untuk kesehatan digunakan untuk pengobatan luka pada kulit tertentu dan lainnya.
- Untuk penyimpanan beras, kacang-kacangan agar terhindar dari kutu (fumigasi)

BAHAYA-BAHAYA TERHADAP KESEHATAN

Gas karbon dioksida normal diudara bebas berkisar 0,035 % (volume), bila lebih akan mengganggu kesehatan. Menghirup karbon dioksida yang berkonsentrasi tinggi berbahaya bagi pernafasan. Pada konsentrasi yang sangat tinggi dapat menyebabkan kehilangan kesadaran dan bahkan kematian.

Dapat mempengaruhi kewaspadaan mental (efek narkotik) dan pernafasan bila konsentrasi didalam ruangan di atas 2% Bila konsentrasinya di atas 7%, kehilangan kesadaran mudah sekali terjadi dengan cepat

Ada resiko akumulasi pada daerah rendah (lubang, gorong-gorong, basemen, dan sebagainya) yang dapat membuat kondisi yang berbahaya

KETAHANAN MATERIAL

Karbon dioksida tidak menyebabkan karat sehingga semua jenis metal biasa dapat dipergunakan, asalkan peralatan dirancang untuk tahan terhadap proses tekanan.

PT. SURYA BIRU MURNI ACETYLENE
Industri Gas Acetylene, Oxygen & Nitrogen

SBM

LEMBAR DATA MATERIAL untuk KESELAMATAN (MSDS)

Karbon Dioksida, gas	MSDS No. 108

SPESIFIKASI TEKNIS

PENYIMPANAN & PENANGANAN BEJANA TEKAN

PENYIMPANAN BEJANA TEKAN

Bejana tekan harus disimpan secara vertikal di tempat yang
sejuk dan berventilasi, jauh dari sumber-sumber panas
atau material yang mudah terbakar
Lindungi bejana tekan ; terutama bagian krannya,
dari kerusakan fisik baik dalam keadaan penuh atau kosong.
Jangan biarkan bagian manapun dan silinder itu terkena
panas di atas 55°C
Periksa apakah bejana tekan telah berlabel.
Untuk bejana tekan yang terisi penuh harus dijaga agar outlet
terpasang pada tempatnya
Tutuplah kran jika silinder kosong

HAL HAL YANG PERLU SEBELUM DIGUNAKAN

Bejana tekan/ krannya tidak boleh terkena minyak atau gemuk.
Tutuplah kran bila tidak digunakan
Selalu gunakan regulator untuk pemakaian sistem.
Bejana tekan tidak boleh tertindih.
Buka/tutup kran bejana tekan pelahan-lahan.

ALAT PERLINDUNGAN DIRI (APD)

Orang-orang yang pekerjaannya berhubungan dengan
pemindahan silinder gas harus dilengkapi dengan:
- sepatu safety
- sarung tangan kulit .
Pakaian kerja dan kacamata pengaman juga dianjurkan.

KEBOCORAN

Pindahkan ke tempat yang berventilasi baik
Hentikan kebocoran bila memungkinkan.
Kosongkan tempat disekitarnya dan arah pergerakan gas
Bila kebocoran tidak dapat dihentikan, pindahkan bejana te-
kan ke tempat yang aman dan biarkan sampai kosong

KEBAKARAN

Karbon dioksida tidak memperbesar pembakaran.
Karbon dioksida merupakan media pemadam kebakaran.
Pindahkan bejana tekan yang secara tidak langsung

PEMBUANGAN GAS

Buanglah sisa gas karbon dioksida keudara pelan-pelan
pada ruangan terbuka dan jauhkan dari orang/pekerja.
Hati hati gas buangan akan sangat dingin

PERTOLONGAN PERTAMA PADA KECELAKAAN

Bila korban sadar:
- pindahkan ke daerah terbuka yang tidak terkontaminasi
  agar dapat menghirup udara segar
- jaga agar tetap hangat dan tenang
  hubungi dokter

Bila korban tidak sadar:
- pindahkan ke daerah yang tidak terkontaminasi dan
  berikan bantuan pernafasan
- bila telah dapat bernafas kembali, lakukan seperti di
  atas. Tindakan selanjutnya harus dilihat dari gejala dan
  pendukungnya.

INFORMASI TAMBAHAN

Informasi, rekomendasi dan data yang ada di lembaran
ini bertujuan untuk memberikan petunjuk dasar kepada
pengguna gas-gas PT Surya Biru Murni Acetylene demi
keselamatan pengguna dalam menangani dan
menggunakan gas-gas tersebut.
Lembar Data Material untuk Keselamatan (MSDS) untuk
gas-gas dan gas-gas campuran yang dikirim oleh
PT Surya Biru Murni Acetylene juga tersedia.
Sangatlah penting demi keselamatan pengguna gas-gas
tersebut agar orang-orang yang menangani gas tersebut
terlatih dengan baik dan benar-benar mengerti kemungkinan
bahaya-bahaya yang dapat terjadi.
Informasi dan saran lebih lanjut mengenai hal-hal yang
berhubungan dengan penanganan atau penggunaan
yang aman dari produk-produk ini dapat diperoleh di
kantor-kantor PT Surya Biru Murni Acetylene terdekat.

Label 2 2 Non flammable, non toxic gas

1 IDENTIFICATION OF THE SUBSTANCE / PREPARATION AND OF THE COMPANY / UNDERTAKING

Trade name	: Nitrogen
MSDS No	: AL089A
Chemical formula	. N2
Company identification	: AIR LIQUIDE SA
	France
	See paragraph 16 "OTHER INFORMATION"
Emergency phone nr	: See paragraph 16 "OTHER INFORMATION"

2 COMPOSITION / INFORMATION ON INGREDIENTS

Substance / Preparation   : Substance.

Substance name	Contents	CAS No	EC No	Index No	Classification
Nitrogen	100 %	7727-37-9	231-783-9	---	

Contains no other components or impurities which will influence the classification of the product.

3 HAZARDS IDENTIFICATION

Hazards identification   : Compressed gas.
In high concentrations may cause asphyxiation.

4 FIRST AID MEASURES

First aid measures
- Inhalation   : In high concentrations may cause asphyxiation. Symptoms may include loss of mobility/consciousness. Victim may not be aware of asphyxiation.
Remove victim to uncontaminated area wearing self contained breathing apparatus. Keep victim warm and rested. Call a doctor. Apply artificial respiration if breathing stopped.

5 FIRE-FIGHTING MEASURES

Flammable class	: Non flammable.
Specific hazards	: Exposure to fire may cause containers to rupture/explode.
Hazardous combustion products	: None.
Extinguishing media	
- Suitable extinguishing media	: All known extinguishants can be used.
Specific methods	: If possible, stop flow of product.
	Move away from the container and cool with water from a protected position.
Special protective equipment for fire fighters	: In confined space use self-contained breathing apparatus.

6 ACCIDENTAL RELEASE MEASURES

Personal precautions
: Evacuate area.
Wear self-contained breathing apparatus when entering area unless atmosphere is proved to be safe.
Ensure adequate air ventilation.

Environmental precautions
: Try to stop release.

Clean up methods
: Ventilate area.

7 HANDLING AND STORAGE

Storage
: Keep container below 50°C in a well ventilated place.

Handling
: Suck back of water into the container must be prevented.
Do not allow backfeed into the container.
Use only properly specified equipment which is suitable for this product, its supply pressure and temperature. Contact your gas supplier if in doubt.
Refer to supplier's container handling instructions.

8 EXPOSURE CONTROLS / PERSONAL PROTECTION

Personal protection
: Ensure adequate ventilation.

9 PHYSICAL AND CHEMICAL PROPERTIES

Physical state at 20 °C : Compressed gas.
Colour : Colourless gas.
Odo(u)r : No odour warning properties.
Molecular weight : 28
Melting point [°C] : -210
Boiling point [°C] : -196
Critical temperature [°C] : -147
Vapour pressure, 20°C : Not applicable.
Relative density, gas (air=1) : 0.97
Relative density, liquid (water=1) . Not applicable.
Solubility in water [mg/l] : 20

10 STABILITY AND REACTIVITY

Stability and reactivity
: Stable under normal conditions.

11 TOXICOLOGICAL INFORMATION

Toxicity information
: No known toxicological effects from this product.

12 ECOLOGICAL INFORMATION

Ecological effects information
: No known ecological damage caused by this product.

13 DISPOSAL CONSIDERATIONS

General : Do not discharge into any place where its accumulation could be dangerous.
To atmosphere in a well ventilated place.
Contact supplier if guidance is required.

14 TRANSPORT INFORMATION

UN No. : 1066
H.I. nr : 20
ADR/RID
- Proper shipping name : NITROGEN, COMPRESSED
- ADR Class : 2
- ADR/RID Classification code : 1 A
- Labelling ADR : Label 2.2 : Non flammable, non toxic gas.
Other transport information : Avoid transport on vehicles where the load space is not separated from the driver's compartment.
Ensure vehicle driver is aware of the potential hazards of the load and knows what to do in the event of an accident or an emergency.
Before transporting product containers :
- Ensure that containers are firmly secured.
- Ensure cylinder valve is closed and not leaking.
- Ensure valve outlet cap nut or plug (where provided) is correctly fitted.
- Ensure valve protection device (where provided) is correctly fitted.
- Ensure there is adequate ventilation.
- Compliance with applicable regulations.

15 REGULATORY INFORMATION

EC Classification : Not included in Annex I.
Not classified as dangerous preparation/substance.
EC Labelling : No EC labelling required.
- Symbol(s) : None.
- R Phrase(s) : None.
- S Phrase(s) : None.

16 OTHER INFORMATION

Asphyxiant in high concentrations.
Keep container in a well-ventilated place.
Do not breathe the gas.
Ensure all national/local regulations are observed.
The hazard of asphyxiation is often overlooked and must be stressed during operator training.

This Safety Data Sheet has been established in accordance with the applicable European Directives and applies to all countries that have translated the Directives in their national laws.

Before using this product in any new process or experiment, a thorough material compatibility and safety study should be carried out.

Details given in this document are believed to be correct at the time of going to press. Whilst proper care has been taken in the preparation of this document, no liability for injury or damage resulting from its use can be accepted.

Recommended uses and restrictions : This SDS is for information purposes only and is subject to change without notice. [Prior to purchase of products, please contact your local AIR LIQUIDE office for a complete SDS (with Manufacturer's name and emergency phone number).]

End of document

AIR LIQUIDE SA
France

Label 2.2 : Non flammable, non toxic gas

Label 5.1 : Oxidizing substances

O : Oxidizing

1 IDENTIFICATION OF THE SUBSTANCE / PREPARATION AND OF THE COMPANY / UNDERTAKING

Trade name	: Oxygen
MSDS No	: AL097A
Chemical formula	: O2
Company identification	: AIR LIQUIDE SA
	France
	See paragraph 16 "OTHER INFORMATION"
Emergency phone nr	: See paragraph 16 "OTHER INFORMATION"

2 COMPOSITION / INFORMATION ON INGREDIENTS

Substance / Preparation : Substance.

Substance name	Contents	CAS No	EC No	Index No	Classification
Oxygen	: 100 %	7782-44-7	231-956-9	008-001-00-8	O; R8

Contains no other components or impurities which will influence the classification of the product.

3 HAZARDS IDENTIFICATION

Hazards identification : Compressed gas.
Oxidant. Strongly supports combustion. May react violently with combustible materials.

4 FIRST AID MEASURES

First aid measures
- Inhalation : Continuous inhalation of concentrations higher than 75% may cause nausea, dizziness, respiratory difficulty and convulsion.
Remove victim to uncontaminated area.

5 FIRE-FIGHTING MEASURES

Flammable class	: Non flammable.
Specific hazards	: Exposure to fire may cause containers to rupture/explode.
	Supports combustion.
Hazardous combustion products	: None.
Extinguishing media	
- Suitable extinguishing media	: All known extinguishants can be used.
Specific methods	: If possible, stop flow of product.
	Move away from the container and cool with water from a protected position.
Special protective equipment for fire fighters	: None.

6 ACCIDENTAL RELEASE MEASURES

Personal precautions	: Evacuate area.
	Ensure adequate air ventilation.
	Eliminate ignition sources.
Environmental precautions	: Try to stop release.
	Prevent from entering sewers, basements and workpits, or any place where its accumulation can be dangerous.
Clean up methods	: Ventilate area.

7 HANDLING AND STORAGE

Storage	: Segregate from flammable gases and other flammable materials in store.
	Keep container below 50°C in a well ventilated place.
Handling	: Use no oil or grease.
	Open valve slowly to avoid pressure shock.
	Suck back of water into the container must be prevented.
	Do not allow backfeed into the container.
	Use only properly specified equipment which is suitable for this product, its supply pressure and temperature. Contact your gas supplier if in doubt.
	Keep away from ignition sources (including static discharges).
	Refer to supplier's container handling instructions.

8 EXPOSURE CONTROLS / PERSONAL PROTECTION

Personal protection	. Do not smoke while handling product.
	Wear suitable hand, body and head protection. Wear goggles with suitable filter lenses when use is cutting/welding.
	Avoid oxygen rich (>21%) atmospheres.
	Ensure adequate ventilation.

9 PHYSICAL AND CHEMICAL PROPERTIES

Physical state at 20 °C	: Compressed gas.
Colour	: Colourless gas.
Odo(u)r	: No odour warning properties.
Molecular weight	: 32
Melting point [°C]	: -219
Boiling point [°C]	: -183
Critical temperature [°C]	: -118
Vapour pressure, 20°C	: Not applicable.
Relative density, gas (air=1)	: 1.1
Relative density, liquid (water=1)	: 1.1
Solubility in water [mg/l]	: 39
Flammability range [vol% in air]	: Oxidiser.
Auto-ignition temperature [°C]	: Not applicable.
Other data	: Gas/vapour heavier than air. May accumulate in confined spaces, particularly at or below ground level.

10 STABILITY AND REACTIVITY

Stability and reactivity
: May react violently with combustible materials.
  May react violently with reducing agents.
  Violently oxidises organic material.

11 TOXICOLOGICAL INFORMATION

Toxicity information
: No known toxicological effects from this product.

12 ECOLOGICAL INFORMATION

Ecological effects information
. No ecological damage caused by this product.

13 DISPOSAL CONSIDERATIONS

General
. To atmosphere in a well ventilated place.
  Do not discharge into any place where its accumulation could be dangerous.
  Contact supplier if guidance is required.

14 TRANSPORT INFORMATION

UN No.
. 1072

H.I. nr
: 25

ADR/RID

- Proper shipping name
: OXYGEN, COMPRESSED

- ADR Class
. 2

- ADR/RID Classification code
: 1 O

- Labelling ADR
: Label 2.2 : Non flammable, non toxic gas.
  Label 5.1 : Oxidizing substances.

Other transport information
: Avoid transport on vehicles where the load space is not separated from the driver's compartment.
  Ensure vehicle driver is aware of the potential hazards of the load and knows what to do in the event of an accident or an emergency.
  Before transporting product containers :
  - Ensure that containers are firmly secured.
  - Ensure cylinder valve is closed and not leaking.
  - Ensure valve outlet cap nut or plug (where provided) is correctly fitted.
  - Ensure valve protection device (where provided) is correctly fitted.
  - Ensure there is adequate ventilation.
  - Compliance with applicable regulations.

15 REGULATORY INFORMATION

EC Classification
: Index No : 008-001-00-8
  O; R8

EC Labelling

- Symbol(s)
. O : Oxidizing

- R Phrase(s)
: R8 : Contact with combustible material may cause fire.

- S Phrase(s)
: S17 : Keep away from combustible material.

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France

16 OTHER INFORMATION

Ensure all national/local regulations are observed.
Ensure operators understand the hazard of oxygen enrichment.

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