

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

From: W. H. Meyer

WHM-89-77

September 12, 1977

Laboratory Department Summary
August, 1977

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From: W. H. Meyer

WHM-89-77

September 12, 1977

Laboratory Department Summary

August, 1977

The Bishop Plant Laboratory activities during August, 1977, are attached. Section I contains a summary of the Analytical Group activities with a table of analytical statistics. Section II contains a Quality Assurance Summary and a table listing shipments, quality waivers, quality complaints and sample shipping statistics. Section III describes the status of Special Problems Group projects by Unit Chemist assignment. A table listing the priorities, justifications and estimated completion dates of current Special Problems projects is also included in Section III.


W. H. Meyer

WHM/jm
Attached Report

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I. ANALYTICAL GROUP

Competitive Evaluation

Methyl Formcel - A sample of Borden-produced 55% Methyl Formcel was obtained from Rohm and Haas. An evaluation report will be issued.

Polyol

TMP - Analytical support was provided for the preparation of Laboratory recrystallized TMP. Samples were sent to Sartomer and PPG for evaluation.

PE AML - The PE AML storage tank number 2458 is now being analyzed daily for sodium formate. These analyses will be used for charging of product transfers to Virginia Chemical Company.

Paraform

Methyl Formcel - Support was provided for efforts to establish minimum concentration of caustic additive necessary to maintain low methylal build in Methyl Formcel product. To date, 100ppm sodium combined with one hour reactor cook time has proven successful. Lower parameters have not yet been tried.

MO Units

MO-III and IV - Analytical support was provided for rate run calculations around these two units. The runs will be repeated due to feed unit (MS) upsets.

Stabilizer - Methocel stabilizer addition tanks and pumps have been re-located. Methocel analysis in sales grade formaldehyde will be performed three per week until conditions stabilize. Current levels range from 3 to 11ppm with a target of 10ppm.

Oxo Area

Propylene - Four Bayport Terminal propylene samples were analyzed for ppm O₂ for the purposes of cross-checking the Bayport unloading and inventory system.

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Utilities and BOD

The Texas Water Quality Board representatives were in the Plant August 9, 1977, for a 24-hour sampling of the two permit outfalls. Samples taken were split between the TWQB consultant, Ernst Davis, and the Plant Laboratory. A letter report will be issued when results from all sources are in hand. Plant Laboratory results indicate all parameters are well within permit levels.

Analytical Statistics

<u>Sample Count</u>	<u>August</u>	<u>August Y-T-D</u>	<u>Previous Y-T-D</u>
Chemical Section Samples	4,334	39,541	32,210
Instrument Section Samples	<u>2,115</u>	<u>18,458</u>	<u>14,971</u>
Total	6,449	57,999	47,181

Technician Overtime:

Chemical	24	559	527
Instrument	<u>50</u>	<u>544</u>	<u>423</u>
Total	74	1,103	950

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II. QUALITY ASSURANCE

DAA - On August 10, 1977, we received word that GATX 69682 loaded with DAA and shipped on July 19 to Alvi, Mexico, arrived with a high color (color unknown). This tank car was a Union Carbide car, and the customer Union Carbide's. The product shipped out at less than 25. Current status in not known.

Methanol - During the month, the Chemcel Laboratory evaluated U.S.I. specifications for methanol as related to the ultraviolet (U.V.) absorbance. Chemcel MS product meets the U.V. absorbance requirements as follows:

Does not exceed 0.60 at 240 mμ; 0.40 at 250 mμ; 0.15 at 260 mμ; and 0.10 from 270 mμ through 340 mμ.

This evaluation was prompted by a recent barge shipment to Bayport which did not pass this special requirement for U.S.I. on methanol. The methanol was found to contain traces of toluene which increased the U.V. absorbance.

Paraformaldehyde Fine Powder

Dycho Co. Inc. (Niota, Tennessee)- The customer complained that the product appeared wet and caused lumping and balling in use. Three lots were involved in the complaint: Lot 2417 (January 10, 1977), Lot 2379 (April 1, 1976) and Lot 2378 (March 27, 1976). The original analyses and the analyses of the retains show that this product met sales specs. Samples representing each of these three lots were returned to us on August 11, 1977. The returned samples were analyzed and found to meet all of our sales specifications, and no evidence of wet powder or lumps was observed. We understand the customer has used the material.

Tennessee Eastman Co. (Kingsport, Tennessee)- The customer complained of difficulties with Paraformaldehyde fine powder shipped to them on June 7, 1977, out of Lot 2442. The complaint involved "balling up" in their machine. They had checked several bags and found lumps the size of gravel. The customer asked that the 200 bags be replaced with a new lot, and this has been done. Analysis of the lot retain and a large sample returned to us met all sales specifications. We checked the sample returned to us from Tennessee Eastman, and found no lumps, as they had reported. The Charlotte Office handled the complaint. Disposition of the 200 bags from Tennessee Eastman is unknown.

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n-Butyl Acetate - Texas Instruments Specification - Work has started on analyzing n-butyl acetate for residue after evaporation by the Texas Instruments method to determine if we can consistently meet their requirements of 20ppm. We feel we must evaluate this further because we now take a residue product as a result of an energy conservation project.

Paraform Flake - Lehn and Fink Products Co. in Toledo, Ohio, complained that the product they have was low in assay. The lots in question are numbers 189-77 and 194-77. The customer reported an 80.8% formaldehyde on these two lots. We feel that this must be an assay problem with them as a value this low would be impossible to flake. A check of the retains of these two lots shows a range of 92-93%, which is normal. The customer has used all this material and has agreed to an exchange of retain samples for an assay check. We have not received the samples.

Methyl Formcel - Recently Uniroyal Chemical at Geismar, Louisiana, has been concerned about the reported formaldehyde value versus the calculated formaldehyde value on the 55% Methyl Formcel + Acetic Acid blends sent to them. As a result of a meeting with Production and Laboratory personnel, a letter (WHM-85-77) of recommendations on the future handling of this type product has been issued.

Sample Shipping and Quality Assurance Statistics follow:

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Sample Shipping and Quality Assurance Statistics

<u>Sample Shipping</u>	<u>August</u>	<u>August Y-T-D</u>	<u>Previous Y-T-D</u>
Domestic	61	785	796
Export	8	77	129
Plastics	4	59	101
Other	10	702	171
Total	83	1,623	1,197
Shipments (Number of Orders)	743	6,762	6,086
Delayed Due to Quality	0	22	25
Quality Waivers	4	33	11
Quality Complaints	4	42	15
Quality Complaints Accepted	0	12	9

NOTE: See attached copies of Quality Complaints and Delayed Shipments due to quality or quality waiver.

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Customer Complaints Due to Quality
August, 1977

<u>Date</u>	<u>Customer/Plant</u>	<u>Product</u>	<u>Tank Car</u>		<u>Complaint</u>	<u>Comments</u>
			<u>Tank Trailer</u>	<u>Subs</u>		
8-11-77	Dycho Co. Inc.	Paraform Fine Powder		Lots 2417, 2379 and 2378	Product Wet Caused Balling	Customer reported balling of product in use. Returned 3 samples representing these lots. Analysis of the 3 samples shows normal pro- duct.
8-10-77	Alvi, Mexico	Diacetone Alcohol		GATX 69682 Shipped 7-19-77	High Color	Customer reported the color was too high to use. Color unknown. Disposition of pro- duct unknown.
8-10-77	Lelo and Fink Toledo, Ohio	Paraform Flake		Lots 189-77 and 194-77	Low Assay	Customer reported the assay to be 80.8. Original analy- sis and retain analysis re- ported normal assay (92-93%). Suspect method of analysis problem with customer.
8-11-77	Tennessee Eastman Kingsport, Tennessee	Paraform Powder		Lot 2442 200 x 50 lb. Shipped 6-7-77	Product Wet Caused Balling	Customer reports they are un- able to use product. Ordered replacement. Disposition of 200 x 50 lb. product from Lot 2442 unknown. Original analy- sis shows spec product (also analysis of returned sample).

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September 12, 1977

Quality Waivers and Shipments Delayed Due to Quality

August, 1977

Date	Product	Customer	Destination	Tank Car		Delay or Waiver	Comments
				Tank Trailer	Solids		
8-08-77	TMP Lot 7707-A1	Ameel Europe, S.A.	Corpus Christi, TX	500-x55		Waiver	Approved by J. C. Scott, New York Office, Water Level at 0.06.
8-10-77	PE-Pure Lot 222 P ₃	Port of Houston	Houston, TX	450x50		Waiver	Approved by J. C. Scott, New York Office, Ash Content = 0.020 (spec = 0.010).
8-10-77	PE-Pure Lot 222 P ₃	Productos Rival, S.A.	Laredo, TX	650x50		Waiver	Approved by J. C. Scott, New York Office, Ash Content = 0.020 (spec = 0.010).
8-18-77	PE-Pure Lot 222 P ₃	Port of Houston	Houston, TX	250x50		Waiver	Approved by J. C. Scott, New York Office, Ash Content = 0.020 (spec = 0.010).

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III. SPECIAL PROBLEMS

Methanol Oxidation, Formaldehyde Blending
A. J. Branecky, J. D. Presley

MO Efficiency Increase with Trace Amounts of Halogenated Hydrocarbon - The data from the methylene bromide and methylene chloride plant tests is being collected and analyzed. A report will be issued this month.

MO Catalyst Reuse - Work will continue as time permits.

MS-II Crude to MO - Awaiting piping modifications for plant trial.

Water Injection to MO - No progress due to priorities.

Pampa Pipeline Grade Methanol to MO - A report was written in August, and will be issued this month (JDP-03-77). As reported in the last monthly summary, the test methanol showed standard micro unit efficiency and conversion. Final analysis shows that the odor causing agent in the Pampa methanol is ethyl acrylate. Further investigation revealed that this compound is not normally found in pipeline grade methanol, and that it was possibly introduced as a contaminant via the sample container used.

The quality of the pipeline methanol appears to be sufficient for use in Bishop MO units. A plant trial is recommended (following the procedure outline in the report) before plant use is accepted.

MO New Catalyst Screening - Further work on spun silver fiber is pending arrival of new samples. A lot of Engelhard standard silver needle catalyst containing 14ppm tin was received recently. The present specification for tin is ≤ 10 ppm. A sample of the lot (Lot 36, July 6, 1977) demonstrated typical new catalyst efficiency and conversion in the micro unit.

A report (JDP-01-77) was issued recommending that, if the catalyst showed standard efficiency and conversion in the process units, the standard specification for tin be raised to 14ppm. Performance of the catalyst will be followed by the Lab.

Reduce Methylal Formation in the MO Distillation Columns - Initial lab work showed the following stability of 50% formaldehyde containing caustic:

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Blank	21+ days
240ppm caustic	21+ days
480ppm caustic	21+ days
720ppm caustic	4 days
960ppm caustic	3 days

Presently, it is believed that significant methylal formation reduction can be achieved by adding caustic in amounts resulting in fewer than 100ppm in the final product. Hence, no problems are anticipated for product stability. No further progress was made due to priorities. Work continues.

MO Unit Rate Runs - The Lab aided Engineering in obtaining necessary data for MO-III and MO-IV rate runs. No significant data was gathered for MO-II due to mechanical difficulties. A report will be issued by Process Engineering.

Nepera Blend - The Lab was asked to determine the feasibility of making a special product blend for Nepera. The blend consists of acetaldehyde, formaldehyde and methanol in the respective molar ratio 1:0.75:0.25. Nepera indicates that almost any concentration of water could be tolerated in the final blend. However, it would be advantageous to both them and us to have as little water as possible. Initial lab work shows that the blend is (by physical appearance) quite stable at room temperature. Gas chromatographic analysis shows, however, that some chemical reactions are taking place. An attempt will be made to quantify this.

Nepera further indicates that they can withstand 1 to 2% total weight per cent variations in the final blend. Hence, side reaction products, if small, might be tolerable within the 1 to 2 per cent limits. Work continues.

Utilities, Raw Materials, BOD
S. G. Smith

Purchase Specification Update - No progress due to priorities.

BOD Unit Analytical Methods Manual - No progress due to priorities.

Anaerobic Pilot Testing of Chemical Waste - An estimate of equipment costs was completed, and a company to build the anaerobic reactor was located. Work on this project is in abeyance until Process Engineering economics are published, and until the decision is made as to which system to use for the proposed BOD expansion. The systems under study are anaerobic, aerobic and the Union Carbide Unox process.

003101

DAA Color Stability - A laboratory simulation of T-285, the DAA heavy ends column, is being conducted to; 1) find optimum operating conditions of the column, and 2) determine if heavy ends (color precursors) are being passed over to the finishing column, T-294, causing color instability of the finished product.

Elimination of Lamella Underflow - No progress due to priorities.

OSHA Monitoring
J. M. Jackson

Samples of formaldehyde in air, received from Ms. M.R. Blankenship (E.I. duPont) have been analyzed and the results returned. This data was obtained for the Test Method Sub-Committee of the SOCMA Task Force, and will be used to evaluate the NIOSH formaldehyde sampling and analytical method.

Duplicate airborne formaldehyde samples were obtained in the Laboratory and Paraformaldehyde Unit for evaluation of the dimedone versus chromatropic acid formaldehyde methods. These samples were at the request of Mr. C.S. Laubly (Corporate Industrial Hygienist). One set of samples was analyzed by the Bishop Lab by the chromatropic acid method. The duplicate set was sent to Gliddin Durkee for the dimedone analysis. A report will be issued when the dimedone results are received.

Opacity of the TMP concentrator exhaust was determined. Follow-up opacities will be determined to verify results, and a report will be issued.

A summary report on OSHA monitoring data is in progress and will be issued soon.

Girdler Unit Monitoring For Nickel Tetracarbonyl
A. R. Hancock

Girdler unit nitrogen purge gas was monitored for nickel tetracarbonyl on August 7, following the planned girdler unit shutdown. No nickel tetracarbonyl was detected. Procedures and results are being reported.

Esters, Methylal, Paraform, Formcels
R. G. Hilbrich, K. B. Benkendorfer

Feed Contaminants and Liquid Paraform Stability - The study has been completed, and a full report is being written.

003102

Paraform Material Balance - Delayed, pending full-time Process Engineering coverage on the Paraform Unit.

Acetate Catalyst Evaluation - Dibutyltin diacetate was evaluated in the laboratory as a potential replacement catalyst for MSA in the Acetate Unit. It was found to be unsatisfactory because of low activity. A report has been written.

Alternate n-Butyl Acetate Process - No progress due to priorities.

Paraform, Formcels and Ester Unit Analytical Manual - No progress due to priorities.

Acetate Unit Support - A study is underway to determine if there is a correlation between the acid concentration in the organic layer and the conductivity of the water layer in the acetate reactor column (T-268) distillate. It is hoped that this will provide a method to continuously monitor the acid level in the organic layer. Numerous samples have been taken and analyzed. Work continues.

1,3-Butylene Glycol, DAA
A. R. Hancock

1,3-BG Unit Support - None required. Unit down.

1,3-BG Cost Reduction - It may be possible to replace the vapor sidestream from the BG finishing column (T-227) with a liquid sidestream or a residue stream. About \$50M per year in steam savings would result. Data from the previous run period was promising. A plant trial will be proposed at the appropriate time, if data continues to indicate feasibility.

DAA Unit Support - No lab support needed.

DAA Laboratory Reactor Study - No progress.

Pentaerythritol, Weak Formaldehyde
H. H. Thigpen, G. R. Stasney

PE Efficiency - No progress.

PE Color - The carbon charged on June 25 continues to remove color satisfactorily. However, there has been some color breakthrough indicated

003103

by detectable amounts of color in the "B" filtrate. A filtrate color of 12 PtCo units showed up in the "B" filtrate on August 8. There has been a very slow increase to 17-18 PtCo units on September 7. Removal will remain satisfactory until the "B" filtrate color increases to 25-30 PtCo units. The PE product will then have a color of about 5 PtCo units (5% solution).

We have produced approximately 680 lbs. of PE per pound carbon on the current charge (through September 7). This is a satisfactory carbon bed life. Previous lab data indicated that a minimum of about 630 lbs. of PE product per pound of carbon would be obtained. Economics were based on that value. Current data from the unit confirms the prediction based on previous lab data.

We will continue to monitor the performance of the carbon bed. A detailed report will be issued after the current charge is replaced.

Lower Sulfate in PE "A" Mother Liquor (AML) - The acid digester temperature and pressure control systems were repaired and the temperature can be maintained high enough (240-250°F) to minimize the amount of sulfuric acid required. During the next month, we will continue efforts to tune this system so we can meet the Virginia Chemical request for 0.5% or less sodium sulfate in the AML sodium formate (dry basis).

Methanol Synthesis

F. R. Dow

Fuel Gas Quality - A sample of fuel gas analyzed August 17 contained 97.3 vol. % methane. This was about 0.5 vol. % higher than the June and July analysis.

Sulfur was not monitored by the Lab because CCCTC was using the Tracor sulfur analyzer. A letter will be issued outlining plans for resolving discrepancies in sulfur analyses between the various process analyzers.

MS-II Analytical Support - DPG, Selas and the combined reformer gases have been analyzed in the lab for comparison with the process analyzer data from the MS-II computer output.

The process analyzer and Lab G. C. analysis of reformer gas composition shows excellent agreement for CO and CH₄, but the Lab shows about 1% more CO₂ than the process analyzer for the reformer streams. This difference is unexplainable at this time. Work will continue.

003104

Oxo Butanol, Propanol
A. J. Bacon

Oxo Reactor Accountability/Stability - Little work was done due to priorities. Use of propylene in heavy ends separation scheme was tried on a larger scale and still looks good, though quantitative data is not available.

A secrecy agreement has been sent to Witco Chemical Co. by J. W. Corley (Houston). Samples for foaming studies will be sent when the agreement has been received.

Oxo, Butanol, Propanol Support - Support work centered around the Oxo run number 3 catalyst dump August 7, and drumming August 8. Sampling during drumming was coordinated and analytical work was carried out. Rhodium accounting and loss analysis reports will be issued in early September.

Minor troubles after startup included uncertainty of reactor level (BuH concentration) and high T-163 overhead rates. We are setting up a unit lab analytical method to determine BuH levels in the reactor. Instrument Lab personnel are running these analyses as needed until this is complete. High T-163 overhead rates could not be traced to any high inerts make through the reaction area. Raising the product separator base temperature seems to have diminished the problem.

Quality Lab analyses of T-67 liquid sidestream product BuOH have shown that project to be a success so far.

T-259 overhead samples were analyzed over approximately 4 days in the latest attempt to improve side fluctuations in daily yield accounting efficiencies in ProOH. Data showed very minor changes in ProOH assay for this stream, revealing that its contribution to any WIP assay changes are minimal.

Process Technology
B. C. Kerr

Technical Report Abstracting - Preparation of technical report abstracts is current. Abstract card files are current through July, 1977.

Production Initiated Process Technology - It was reported last month that a simplified reporting form had been submitted to Production for comments and revisions. Only one report of six sent out has been returned with comments.

003105

Chemcel Plant Process

Technology Review for New Employees - A booklet has been prepared containing a short review of Chemcel unit operations. Simplified flow diagrams, process chemistry and other information is presented for each of the process units and the BOD system. Copies are available in Process Engineering and Plant Engineering and the Laboratory.

Miscellaneous

Jim Jackson and Gary Boyd attended a meeting in Houston to establish priorities on Chemical Company streams to be subjected to toxicity testing.

Susan Smith reported as a Chemist to Special Problems on August 15. Her unit assignment will be Utilities, Raw Materials and BOD.

Leanne Mayerle transferred from CCCTC to Chemcel as a Special Problems Chemist, effective September 1, 1977. Her assignment will be the TMP Unit.

Bobby Briell accepted employment as a Chemist in the Special Problems section effective September 6, 1977. His unit assignment will be PE.

Temporary summer employees, K. B. Benkendorfer, Mauro Castro and Randy Shafer, returned to their respective colleges during the month.

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Mo Efficiency Improvement With Trace Amounts of Halogenated Hydrocarbons	Branecky, Presley	1	Raw Material Savings	Plant Testing Completed by September 1, 1977
MO New Catalyst Screening		2	To Establish Alternate Suppliers, and Improve Catalyst Quality	Continuing
MO Catalyst Reuse		2	Reduce Silver Inventory	December 1, 1977
MS-II Crude to MO		1	Steam Savings	Plant Testing Completed by November 1, 1977
Water Injection to MO		2	Steam Savings	December 1, 1977
Reduce Methylal Formation In the MO Distillation Columns		1	Raw Material Savings	Plant Testing by January 1, 1978
Nepera Blend		1	New Sales	Lab Work Completed by November 1, 1977
BOD Analytical Methods Manual	Smith	2	Maintenance of Business	December 31, 1977
Anaerobic Pilot Testing of Chemical Waste Streams		1	To Test Anaerobic Process as Alternate BOD Expansion	Undetermined
Purchase Specification Update		2	Maintenance of Business	May 1, 1978
Elimination of Lamella Underflow		2	WTFF Objective	May 1, 1978

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
DAA Color Stability	Smith	1	Cost Reduction	November 1, 1977
OSHA Monitoring	Jackson	1	Ensure Employee Health and Safety, OSHA Compliance	Continuing
Paraform Material Balance	Hilbrich	2	Possible Raw Material Savings	November 1, 1977
Alternate n-BuAc Process		2	Steam Savings	January 1, 1978
Paraform, Formcels, Esters Unit Analytical Methods		1	Maintenance of Business	Continuing
Acetate Unit Support		1	Maintenance of Business	Continuing
Alternate TMP Purification System	Mayerle	1	Raw Material Savings	January 1, 1978
TMP Unit Support		1	Maintenance of Business	Continuing
1, 3-BG Unit Support	Hancock	1	Maintenance of Business	Continuing
1, 3-BG Cost Reduction		1	Cost Reduction	October 1, 1977
DAA Unit Support		1	Maintenance of Business	Continuing

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
DAA Laboratory Reactor Study	Hancock	1	Increase Unit Capacity and Steam Savings	November 1, 1977
PE Efficiency	Briell	1	Raw Material Savings Plus Increased Capacity 4-8% Potential Increase	December, 1977
PE Unit Support		1	Maintenance of Business	Continuing
Lower Sulfate in PE AML		1	Comply With Virginia Chemical Company Request	October 1, 1977
PE Color (Evaluate Carbon Bed Performance)		1	Maintenance of Business	Continuing
Fuel Gas Quality	Dow	2	Improve Fuel Gas Accountability	Continuing
MS-II Analytical Support		1	Maintenance of Business	Continuing
Nepera Blend Analytical Support		2	New Sales	Laboratory Work Completed by November 1, 1977
DAA Laboratory Reactor Study Analytical Support		2	Increase Unit Capacity and Steam Savings	November 1, 1977
Oxo Reactor Accountability/Stability	Bacon	1	Improve Reactor Control	Continuing
Oxo, Butanol, Propanol Support		1	Maintenance of Business	Continuing

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Rhodium Accounting	Bacon	1	Maintenance of Business Catalyst Savings	Continuing

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